THE JOURNAL
OF THE
AMERICAN LEATHER CHEMISTS ASSOCIATION

VOL. XLVIII APRIL 1953 No. 4

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GOLDEN ANNIVERSARY JUNE 7th – 10th 1953 ALCA FOUNDED 1903 CINCINNATI, OHIO MEETING

SUBSCRIPTION: Members $12.00 a Year Non-Members $15.00

Published Monthly by

THE AMERICAN LEATHER CHEMISTS ASSOCIATION

PUBLICATION OFFICE, EASTON, PA.

Entered as Second-Class Matter at Post Office, Easton, Pa.
Acceptance for Mailing at Special Rate of Postage as Provided for in Section 1103.
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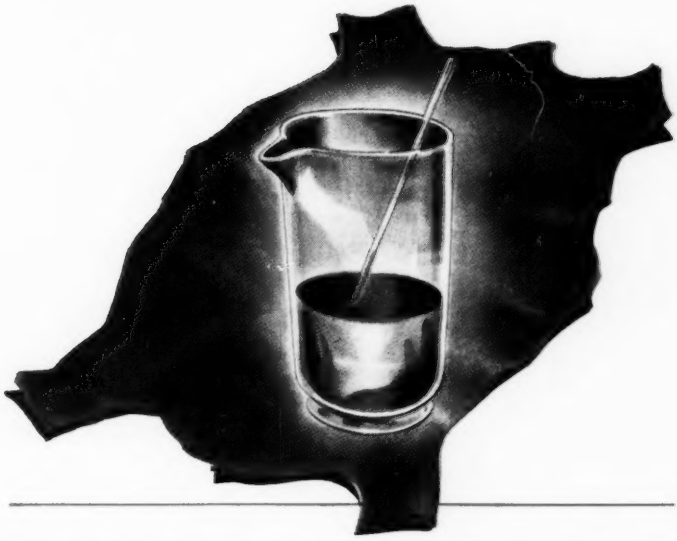

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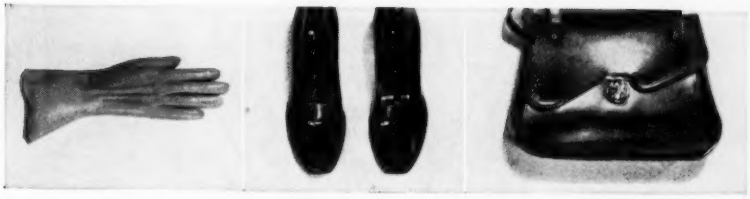

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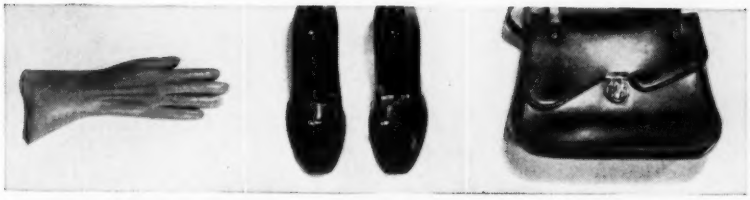

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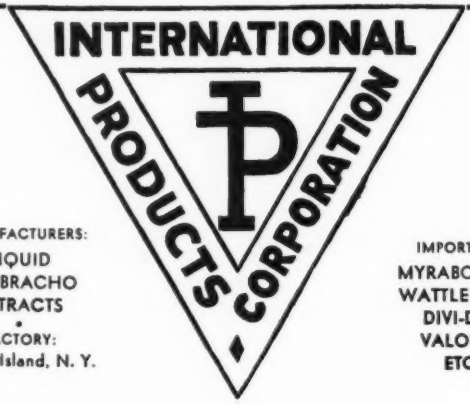

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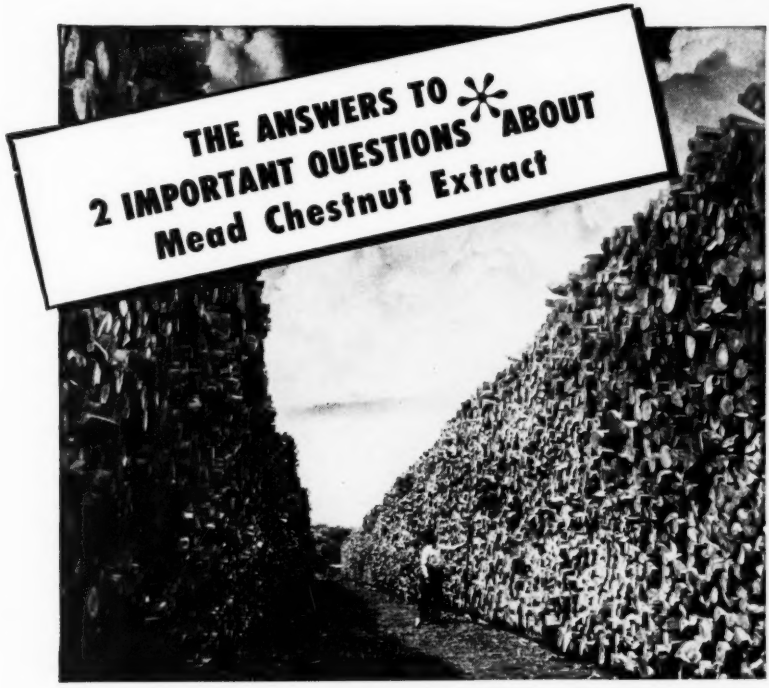

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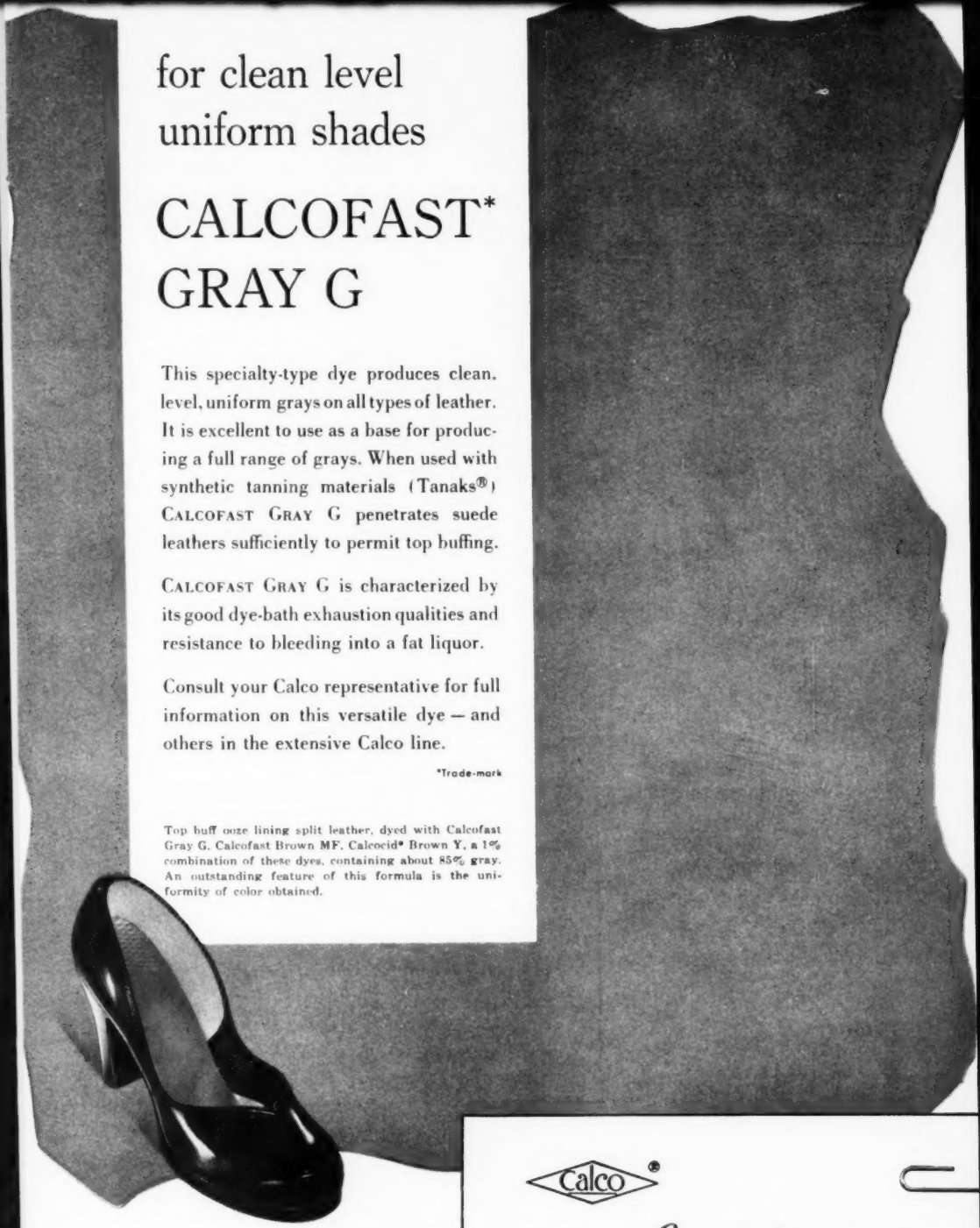

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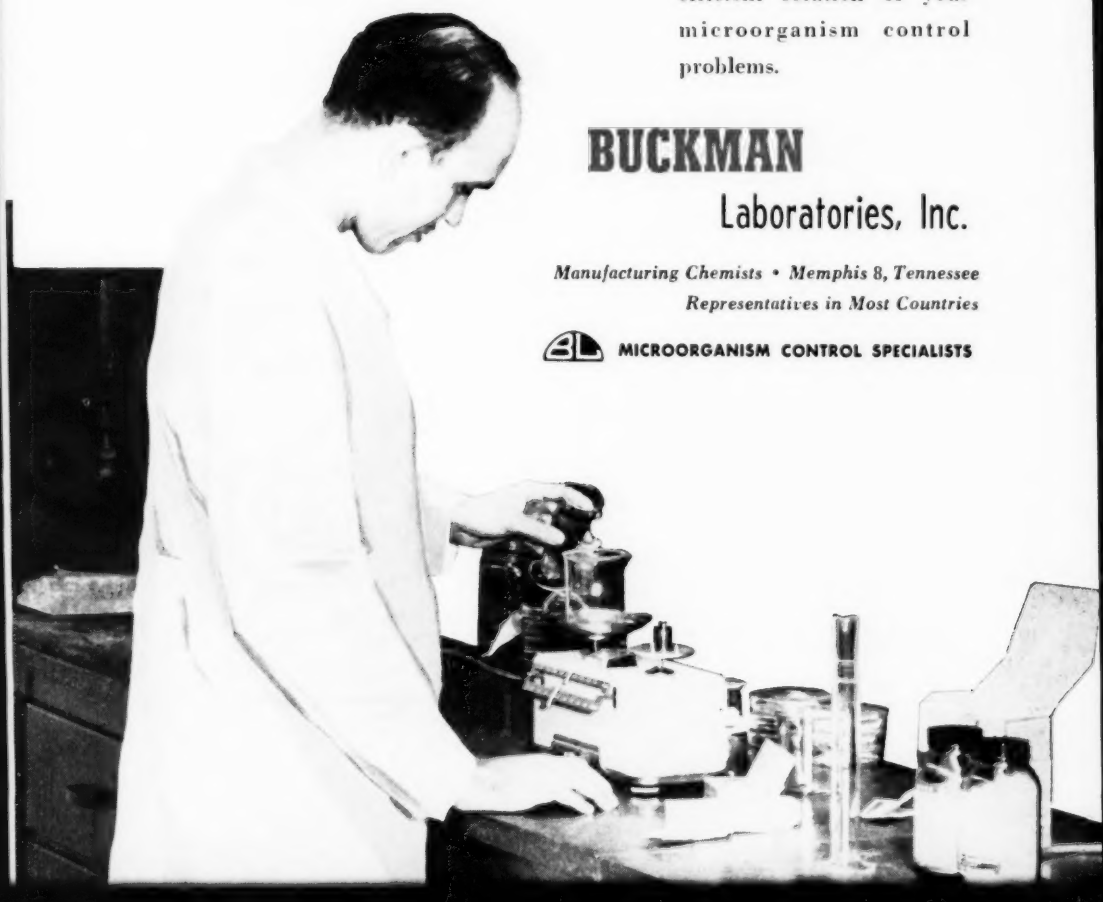

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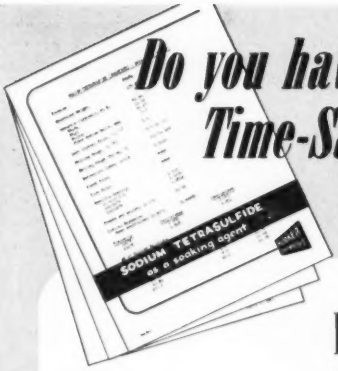

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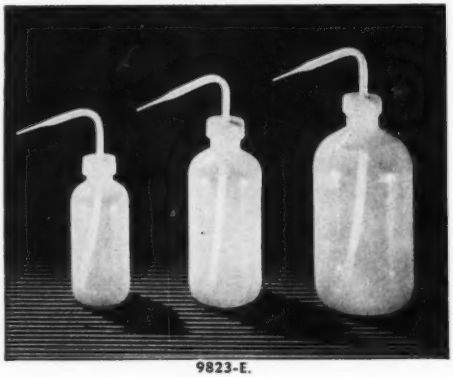

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Checks for subscriptions and advertisements should be made payable to the American Leather Chemists Association.

Subscriptions—Members, $12.00 annually; Non-members, $15.00 annually.

Published monthly by the American Leather Chemists Association.

Entered as Second-class Matter at the Post Office at Easton, Pa.

Acceptance for mailing at special rate of postage provided for in section 1103, Act of October 3, 1917, authorized July 16, 1918.

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SECRETARY'S NOTICE

International Union of Leather Chemists Societies has changed the dates of the Barcelona, Spain, meeting.

Dr. A. Harvey, Honorary General Secretary of the International Union of Leather Chemists Societies, has announced that because the original dates of September 13th–18th conflicted with the Semaine du Cuir de France, President Mallebay of the I.U.L.C.S. has changed the dates to September 6th–11th, 1953.

CORRECTIONS

K. H. Gustavson has noted the following corrections to be made in his paper "Reactions of Polyamides with Tanning Agents" which appeared in the November 1952 Journal.

Page 701, line 8 from the bottom, read "chromium"; line 12 from the bottom "that" instead of "than"; line 14 from the bottom, read "esterified". Page 702, line 9 from the top, delete "protein". Page 703, line 3 from the top, read "polyamide"; line 10 from the top "Igamid" instead of "Igamide"; line 11, "Anilin" instead of "Analine"; line 12, "adipic acid and hexamethylene-diamine" instead of "adipic acid-hexamethylene", and "caprolactam" instead of "coprolactam"; line 13 from the bottom, "0.05 meq." instead of "0.5 meq.". Page 704, line 19 from the top, insert reference "31" after "matter". Page 706, line 10 from the top, "Amberlite" instead of "Amerlite". Page 707, line 7 from the bottom, "formate" instead of "formiate". Page 710, in Acknowledgment, read "gratefully". In Table I, No. 2, "Phthalato-Cr-sulfate, 1M phthalate per 1M Cr_2O_3" instead of "Phthalato-Cr-sulfate, 1M Cr_2O_3". The words "ionic" and "non-ionic" may take the place of "polar" and "non-polar" respectively on page 700, in order to conform with the character of the bonds.

ADDENDUM

THERMAL STABILITY OF LEATHER

DECEMBER, 1952

Since the above article appeared in the December, 1952 issue of the Journal, two British Patents No. 573026, November 2, 1945 and No. 587005, April 10, 1947 on "Process for Treating Leather" were called to the attention of the authors. The patents relate to processes for the manufacture of leathers which have outstanding resistance to shrinkage when exposed to heat and cold. The patents cover in part some of the work reported as to prevention of shrinkage by heat treating in air or mineral oil.

REPORT OF HIDE POWDER COMMITTEE

A sample of hide powder furnished by Frank F. Marshall, Ridgway, Pennsylvania and representing a batch designated as Lot No. 26 has been tested by the A.L.C.A. Hide Powder Committee. The average values found by the Committee members in certain comparative tests of A.L.C.A. American Standard Hide Powder, Lot No. 25, and the proposed Lot No. 26 are as follows:

Hide Powder Lot	No. 25	No. 26
Date of testing	Dec. 1952	Dec. 1952
Water soluble content, Blank test residue (grams)	0.30	0.27
Ash, %	0.18	0.21
pH	5.20	5.33
Nontannin grand average, %	19.78	19.77

The Committee has approved Batch No. 26 and has authorized Frank F. Marshall to issue the hide powder in this batch as A.L.C.A. Official American Standard Hide Powder, Lot No. 26. It was declared official January 30, 1953.

G. V. Downing — F. F. Marshall*
E. S. Flinn — G. W. Schultz
A. N. Kay — I. D. Clarke, *Chairman*

*Participated in the testing but not in the approval of the hide powder as official

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Revision of the Physical Testing Methods

By L. Maeser, *Chairman*

In 1948, a joint committee was appointed by the Federal Specifications Board and the A.L.C.A. to revise and rewrite the existing Physical Testing Methods for Leather, and if possible, work out a single set of methods acceptable to both groups. The members of the committee were Robert B. Hobbs and Jerome S. Rogers, representing the Federal Specifications Board, and J. H. Gregory, Fred O'Flaherty, and E. B. Thorstensen, chairman, representing the A.L.C.A.

The committee worked up the requirements of equipment and procedure and a general form of presentation for all the methods which were recognized by the Federal Specifications Board and the A.L.C.A.

The results of the work done by the joint committee were reviewed by the Physical Testing Committee at its meeting in June, 1952. The few changes necessary to bring the methods of the Association in line with the recommendations of the joint committee were accepted.

An editorial subcommittee was appointed to rewrite the Physical Testing Methods of the Association to include the recommendations of the joint committee. The members of this committee were J. S. Rogers, chairman, Charles W. Mann, and E. G. Railo. The subcommittee has completed its work. The revised methods are published in the Journal for the consideration by the Association, as replacements for the existing Physical Testing Methods. This revision has been very complete and major changes have been made in each method. The Physical Testing Committee recommends that all revised methods be given provisional status.

A revision of several of the methods has not been made because more committee work is required before a satisfactory method can be written.

Sampling Methods for Leather for Physical Tests*

SAMPLING LIGHT LEATHER (S1)

1. Sampling for Compliance with a Contract

A. Number of Units to be Sampled

Fifteen test units shall be selected at random for test from each lot. For lots consisting of less than fifteen units, each unit shall be sampled for test.

B. Location and Size of Cuttings

For Hides, Skins and Other Units Except Bellies and Double Shoulders. Cut the test specimen approximately square and as large as needed with one edge parallel and 2 inches from the backbone, beginning 5 inches from the root of the tail. See Figure I, test area W.

For Bellies. Cut the test specimen, X, 2 inches wide and as long as needed with one long edge parallel to and one inch from the belly line JE in Figure I. The middle of the specimen should be located midway between J and E.

For Double Shoulders. Cut the test specimen, Y, 2 inches wide and as long as needed with one long edge parallel to and one inch from the line BE. The middle of the specimen should be located midway between B and E.

2. Sampling for Research and Development Tests

For tests carried out in connection with research or development work, the following sampling procedure is recommended.

A. If one or more treatments are to be compared with a control (untreated, or treated by standard procedure), an equal number of test pieces should be selected for each treatment and for the control.

* All of the following Sampling Methods are Provisional as of February, 1953, except S1 which is a Proposed Method as of the same date.

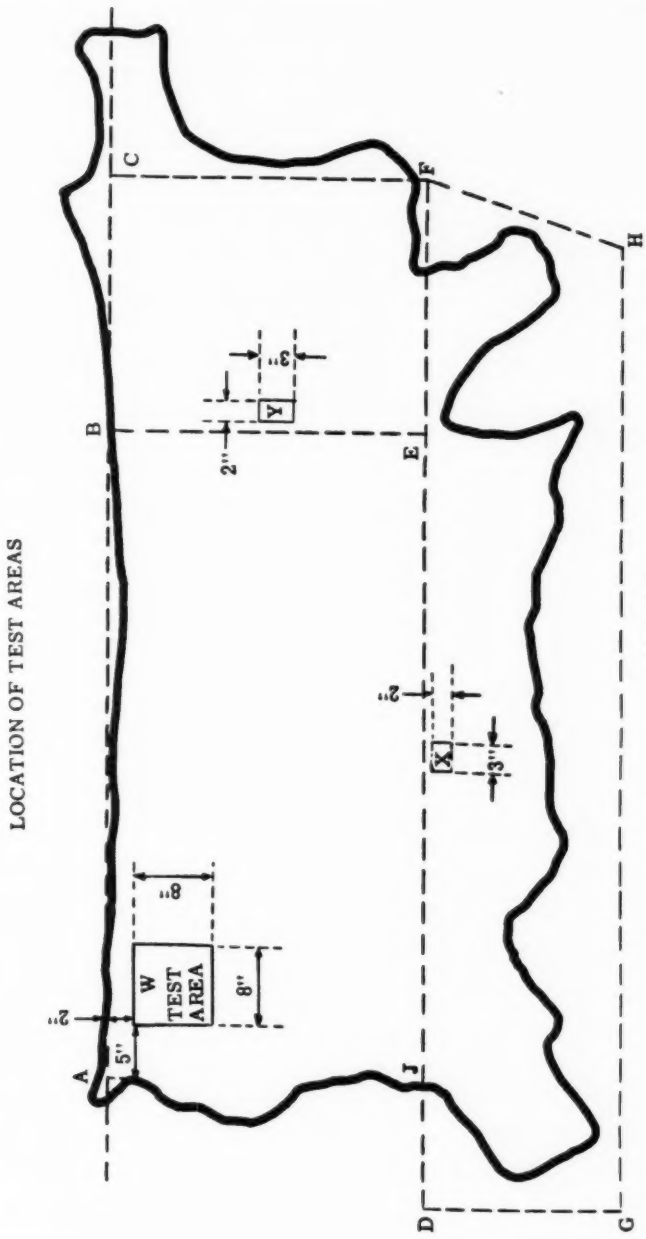


FIGURE 1.

B. The stock from which all test pieces are taken should be as uniform as possible, for example, a single drum load or one day's production, etc.

C. The allocation of the test pieces to the various treatments and control should be done according to a random scheme.

D. Only one treatment should be applied to any one hide.

E. All test pieces should be taken from the same hide location. For optimum test locations and directions of test see Reference 1, "Sampling of Side Upper Leather, No. II", *J.A.L.C.A.* **47,** 352 (1952).

F. The number of hides required for each treatment will depend on the following factors.

1. The smallest treatment effect that the experiment is intended to detect.
2. The probablity with which this detection is to be made.
3. The variability of the property investigated for hides similarly treated.

The investigator should decide on F1 and F2 before carrying out the experiment, and should obtain values for F3 from available tables such as, for example, Table IV of Reference 1. The following formula may be used to calculate the number of hides to be used for each treatment.

$$n = \frac{2\,(A + B)^2 C^2}{D^2} \qquad (1)$$

where

n = number of hides for each treatment (and for control)

A = a factor related to the risk that chance effects might indicate results that would not be confirmed in large scale production (see Table I).

B = a factor related to the risk that the experiment will fail to show real effects of treatments (see Table I).

C = per cent coefficient of variation between hides similarly treated, for the physical property investigated. (See Ref. 1)

D = the smallest treatment effect, expressed as a percentage change in a particular property, that the experiment is intended to detect.

TABLE I

Probability of Finding a Treatment Effect That Does Not Really Exist	Make A Equal To	Probability of Detecting the Selected Minimum Treatment Effect (D%)	Make B Equal To
15.0%	1.03	50%	0.00
10.0	1.28	80	0.84
5.0	1.64	90	1.28
2.5	1.96	95	1.64

Example of Application of Formula (1)

Suppose that two experimental tanning procedures X and Y are to be studied together with a standard tanning procedure S. Let tensile strength be the property investigated. The first step is to find the variability of this test. Ref. (1) shows that a good hide location for this property is block 5, for which the coefficient of variation is 11%. Thus the value for C is 11.

Suppose that the investigator does not wish to be misled by chance effects more than 10 per cent of the time, in finding differences that do not really exist. Table I shows that to this effect the value of A = 1.28.

Furthermore, let it be required that a real treatment effect (improvement in tensile strength) of 15 per cent be detected with a probability of 80 per cent. Thus, the value of D = 15 and Table I gives for B the value 0.84.

Consequently, formula (1) yields

$$n = 2\,\frac{(1.28 + 0.84)^2 (11)^2}{(15)^2} = 4.8 \text{ or } 5 \text{ hides}$$

Thus the experiment shall consist in taking 15 hides and allocating at random 5 hides to X, 5 hides to Y, and 5 hides to S. One tensile specimen will then be cut from each hide in location 5 and the averages of 5 values corresponding to X, Y and S shall be compared. If, for example, the average for X is 3600 lbs/in^2, 3150 lbs/in^2 for Y, and 3000 lbs/in^2 for S, the treatment effects are

600 lbs/in^2 for X
150 lbs/in^2 for Y

To test the significance of these values we form the ratio

$$t = \frac{\text{treatment effect}}{\sqrt{\frac{2}{n}\ \frac{(C)}{(100)}}\ \text{(average of S)}}$$

In this case

$$\text{For treatment X } t_x = \frac{600}{\sqrt{\frac{2}{5}\ (0.11)\quad (3000)}} = 2.88$$

$$\text{For treatment Y } t_y = \frac{150}{\sqrt{\frac{2}{5}\ (0.11)\quad (3000)}} = 0.72$$

Now compare these values to the selected value of A, i. e., 1.28.

t_x is larger, therefore the effect of X (600 lbs/in^2 improvement) is considered real.

t_y is smaller, therefore the effect of Y is not considered significant.

SAMPLING HEAVY LEATHER (S2)

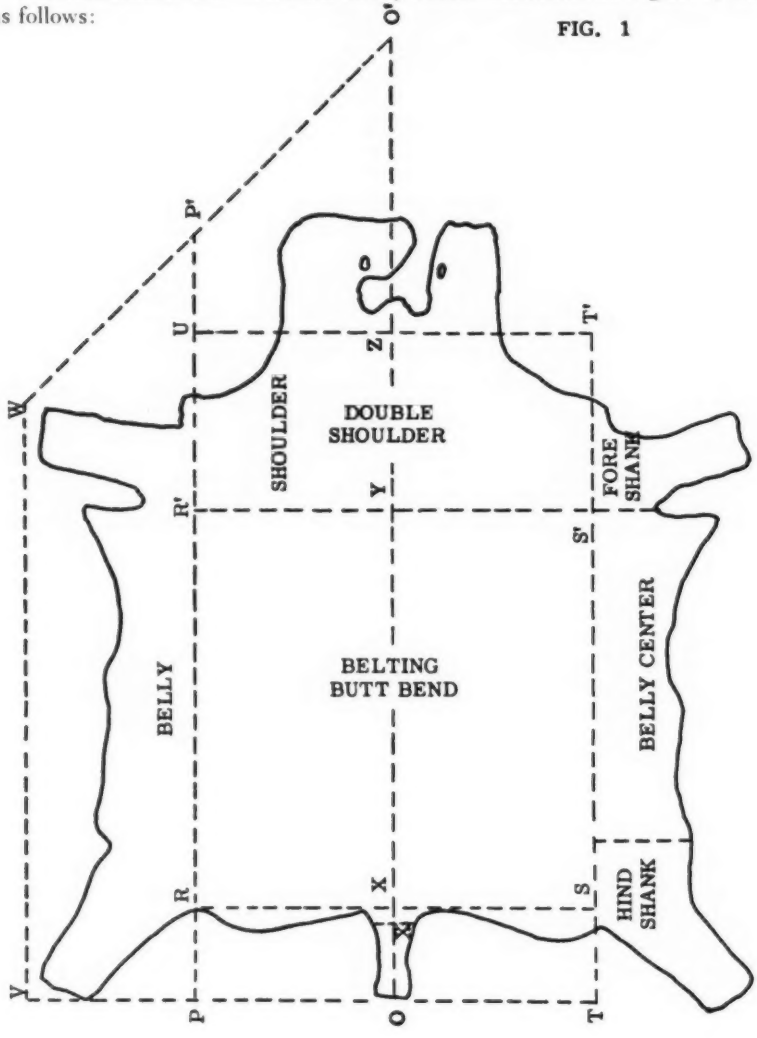
1. Scope

This procedure is intended for use in sampling all types of heavy leather made from hides, sides, or other commercial cuts.

2. Definitions of Hide Parts

The commercial cuts of tanned heavy hides, illustrated in Figure 1, are as follows:

FIG. 1



A Side—OO′WV—is half a hide cut along the backbone line.

A Crop—OO′P′P—is a side with the belly trimmed off.

A Back—OZUP—is a crop with the head trimmed off behind the horn holes.

A Bend—OYR′P—is a "back" with the shoulder cut off at right angles to the backbone line at the break of the fore flank.

A Belting Butt—RUT′S—is a double "back" with the tail cut off at the butt line.

A Belting Butt Bend—RR′S′S—is a double "bend" with the tail cut off at the butt line.

A Sole Leather Butt Bend—PR′S′T—is a double "bend".

A Double Shoulder—R′UT′S′—is obtained when a "belting butt bend" is cut from a "belting butt".

A Belly—VWP′P—is that part of the hide below the belly line (RU).

For steerhide leather, RU passes through a point at or above the top of the front "break" and a point not more than ¾ inch below the top of the rear "break". For cowhide leather, the belly line RU passes through a point at or above the top of the front "break" and a point not more than 2½ inches below the top of the rear "break".

3. Sampling Procedure

The locations from which specimens (or test pieces) are to be cut are indicated in Figure 2.

For hides, sides, crops and backs: The test pieces shall be cut approximately square, as large as needed for the tests to be made, and with one edge parallel to and 1 inch from the backbone line. Cut from the tail to head beginning at a point distant from the root of the tail (X′), 1/6 the distance from the root of the tail to the neck line (1/6 X′Z).

For bends and sole leather butt bends: The test pieces shall be cut approximately square and as large as needed with one edge parallel to and 1 inch in from the backbone line. Cut from the tail to head beginning at a point distant from the root of the tail (X′), 1/4 the distance from the root of the tail to the shoulder line (1/4 X′Y).

For belting butts and belting butt bends: The test pieces shall be cut 2 inches wide the full length of the butt or bend so located that the center line of its long dimension is parallel to the backbone and half way ± 1 inch between the backbone line and the belly line. Cut specimens for physical tests from this strip beginning at a location 1/4 the distance from the butt line RS to the shoulder line R′S′ (1/4 XY) and extending forward as required to obtain the desired number of test pieces.

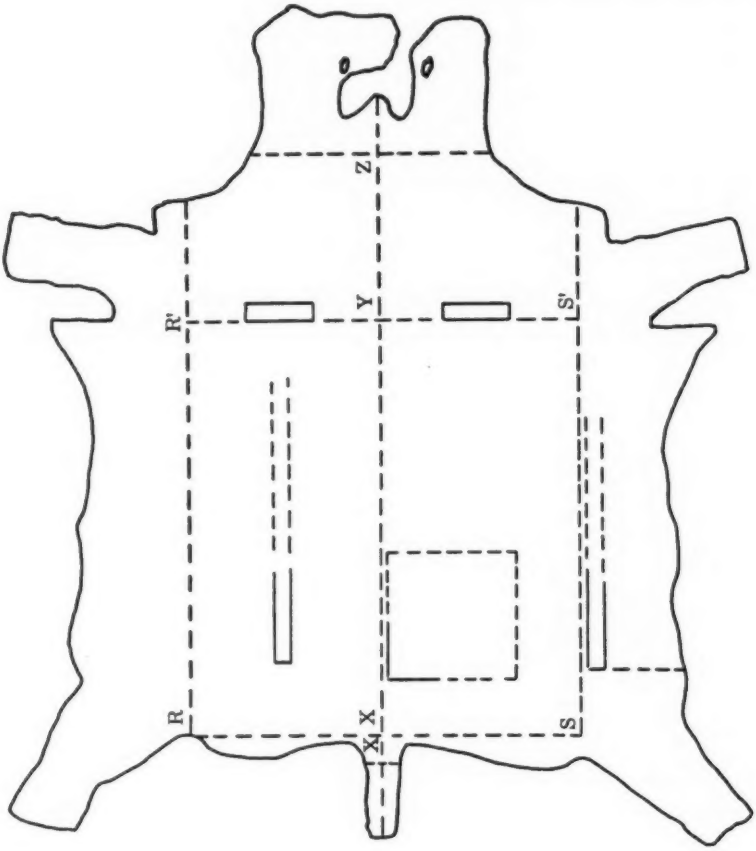


FIG. 2

For bellies: The test pieces shall be cut 2 inches wide and as long as needed with one long-dimension edge parallel to and 1 inch from the belly line, SS′, and in such manner that its end nearer S falls on the line dividing the hind shank from the belly center.

For double shoulders: The test piece (or two if more sample is required) shall be cut 2 inches by 8 inches with one long-dimension edge falling on the line dividing the shoulder and the bend and located equi-distant from the backbone line and the belly line.

4. History and Status of Sampling Method

This method was adopted as official in 1942. The Physical Testing Committee approved parts 2 and 3 October 14, 1947, as changes in detail only and submitted to Council for approval and adoption in 1948 (see JALCA 1948, pg. 9–12).

CONDITIONING LEATHER (S3)

Conditioning shall consist of storing the units or test specimens in the standard atmosphere until they reach weight equilibrium.

1. Scope

This method is suitable for all units and specimens of leather.

2. Specimens

Either the prepared test specimen or the unit from which it is to be cut may be conditioned.

3. Apparatus

A room or chamber in which the standard atmosphere is automatically maintained shall be used for conditioning all specimens.

Standard Atmosphere. The Standard atmosphere shall be air at 73.5° F. ± 2° F. (23° C. ± 1° C.) with a relative humidity of 50% ± 4%. It is intended that the conditioning room or chamber be maintained as close as possible to 50 per cent relative humidity and 23° C. The indicated tolerances are meant to cover variations from these conditions which may occur over short-time cycles in various parts of the room or chamber.

This atmosphere may be obtained by the use of an air-conditioned room or a small chamber such as a desiccator with a saturated solution of $Mg(NO_3)_2.6H_2O$, a saturated solution of $Ca(NO_3)_2.4H_2O$, or a 52 per cent solution of sulfuric acid. (Atmospheres maintained with a salt solution will not approach the standard unless the temperature is near 23° C.)

4. Procedure

All test specimens or the units from which they are to be cut shall be separated, supported so that essentially all surfaces are uncovered, and stored in the standard atmosphere until they reach weight equilibrium. This usually requires from 24 to 75 hours depending on the leather and its thickness. (If forced circulation of the air is not used in the chamber, longer times to reach equilibrium will be required.) The specimens shall have reached equilibrium when hourly weighings show progressive weight changes less than ± 0.1 per cent.

All dimensions of test specimens shall be measured after conditioning. If the units are conditioned, all specimens shall be cut from the unit and measured in the standard atmosphere.

All physical tests shall be made in a room maintained at standard condition. (When specimens are conditioned in a chamber, they shall be removed one at a time and tested immediately. The test shall be completed in less than 10 minutes after removal of the specimen from the chamber.) The conditioning room may be used for both conditioning and testing.

5. Results

The results of any physical test shall state whether or not the specimen was conditioned and tested in the standard atmosphere.

6. History and Status

A standard atmosphere of 65 per cent relative humidity and 70° F. was made official in 1942. This was changed to 73.5° F. ± 2° F. (23° C. ± 1° C.) and 50% ± 4% relative humidity in June 1949.

Physical Testing Methods for Leather

AREA (P1)

1. Scope

This method is intended for use in determining the area of units of all types of leather. It is not intended for measuring the area of physical test specimens.

2. Specimen

The specimen shall be the full unit.

3. Apparatus

The apparatus shall consist of a sheet of transparent, flexible material which has been ruled in square inches. The sheet shall be large enough to cover the specimen completely.

4. Procedure

The specimen shall be spread out on a flat surface. The graduated sheet shall be spread smoothly over and in contact with it. The area shall be determined by counting the number of square inches required to cover the specimen. The area represented by parts of squares not completely over the specimen shall be estimated.

5. Results

The measured area of each unit shall be calculated to the nearest 0.25 square foot.

* All of the following Methods are Provisional as of February, 1953.

The total area of the leather in a sample shall be the sum of the areas of the individual units.

6. History and Status

The planimeter method of measuring the area of skins or large pieces of leather was adopted as official in 1941.

NOTE: The following methods for measuring the area of leather units are not official methods of the Association but are suggested as ways in which the area can be determined with a satisfactory degree of accuracy when a graduated sheet is not available.

WEIGHT METHOD

Apparatus: The apparatus shall consist of the following.

1. A sheet of paper of uniform thickness at least as large as the specimen to be measured.
2. Instrument for cutting paper.
3. Balance and set of weights.
4. Rule or tape graduated in $^1/_8$ inch.

Procedure: A piece of paper, larger than the unit to be measured, shall be spread out on a smooth, flat surface. The unit shall be spread on the paper and a piece of paper the shape and size of the skin shall be cut. The unit-shaped paper shall be weighed on a balance to the nearest 0.1 gram, and the weight recorded as W_1. A rectangular piece of the same paper with an area more than one-half of the total area of the unit shall be cut, weighed, and the weight recorded as W_2. Its dimensions shall be measured to the nearest $^1/_8$ inch and its area computed and recorded as A_2. The area A_1 of the unit shall be calculated to the nearest 0.25 square foot as follows.

$$A_1 = \frac{W_1}{W_2} \times \frac{A_2}{144} \text{ sq. feet.}$$

W_1 is the weight of the skin-shaped paper in grams.
W_2 is the weight of the paper rectangle in grams.
A_2 is the area of the paper rectangle in square inches.

Planimeter method (an exact method for measuring the area of units which will lie flat.)

Apparatus: A planimeter with the following characteristics shall be used.

1. A tracer arm at least 40 inches long,

2. A measuring wheel graduated to read one-fourth of a square foot and with a capacity of 10 square feet and
3. An auxiliary wheel to give the instrument a total capacity of at least 40 square feet.

Procedure: The specimen shall be spread out on a flat surface and the area shall be measured with the planimeter to the nearest one-fourth of a square foot.

THICKNESS OF UNITS (P2)

A unit is an item of leather in its commercial form such as a skin, side, bend, belly, cut sole or block.

1. Scope

This method is intended for use in measuring the thickness of units of all types of leather. It is not intended for use in measuring the thickness of test specimens.

2. Specimen

The specimen shall be the full unit.

3. Apparatus

The Apparatus Shall Consist of

A. A *spring-type gage* graduated in 0.1 milimeter and ½ ounce having a flat presser foot 0.31 ± 0.01 inch in diameter, and a flat anvil 0.31 ± 0.01 inch in diameter. The spring shall exert a force of 1 pound on the foot when the gage reads 2 ounces, and 2 pounds when the gage reads 12 ounces. (The "Woburn" type gage meets these requirements. On this gage 1 ounce equals $^1/_{64}$ inch.) When readings in irons are required using this gage, readings are to be taken in ounces and converted to irons by multiplying by 0.75.

B. A *standard wedge-type gage* having one leg graduated in ½ iron (one iron equals $^1/_{48}$ inch.)

4. Procedure

Light Leathers.

The portion of the specimen to be measured shall be placed between the anvil and presser foot of the spring type gage in such a manner that the specimen is in contact with the whole area of the anvil. With the specimen held in this position, the presser foot shall be lowered gently (not dropped)

until it rests on the specimen. The thickness shall be read from the scale to the nearest 0.1 millimeter, or ½ ounce.

If the dimensions of the specimens are such that the gage will not reach from the edge to the point at which the thickness is desired, the specimen may be folded upon itself with the flesh inside. The thickness of the folded specimen shall be measured, and one-half of this value shall be taken as the thickness. The thickness shall be measured at not less than 5 equally spaced places along and approximately 6 inches from the backbone.

SOLE AND OTHER HEAVY LEATHERS.

The "cut" edge of the specimen shall be inserted between the legs of a standard wedge-type gage so that the plane of the leather is perpendicular to the plane of the gage and bisects the angle formed by the legs. The gage shall be pushed over the edge of the leather to make firm contact with the leather but shall not cause any visible deformation of the specimen. The thickness shall be read from the scale to the nearest ½ iron at the point where the specimen contacts the legs of the gage.

The thickness shall be measured in at least 5 places equally distributed along the "cut" edge of the specimen.

5. Results

The median of the measurements taken on a unit shall be used as its thickness. (Median: If the numerical values of the measurements are arranged in ascending or decending order, the median is obtained as follows: (a) if the number of values is odd, the median is the value in the middle of the series, (b) if the number of values is even, the median is the arithmetic average of the two middle values.)

The medians of all the units measured shall be averaged to obtain the thickness of the sample.

The range and average of the medians shall be reported.

6. History and Status

This method was part of a method for measuring the "Dimensions of Leather" which was adopted as official in 1941.

THICKNESS OF SPECIMENS (P3)

1. Scope

This method is intended for use in measuring the thickness of test specimens in which the dimensions of the specimens are used directly in determining the results of the tests.

2. Specimens

The specimen shall be a piece of leather which has been conditioned and cut to the dimensions required for the particular physical test.

3. Apparatus

The apparatus shall be a gage which is equipped with a dial micrometer, a flat anvil, and a presser foot loaded by dead weight. (The Randall-Stickney dead-weight type of gage meets these requirements.)

The dial shall be graduated to read the distance between the anvil and presser foot to the nearest 0.001 inch.

The anvil shall be flat. Its diameter shall be between 0.375 and 0.385 inch.

The presser foot shall have a flat contact surface, 0.375 ± 0.001 inch in diameter. It shall be dead-weight loaded to exert a total load of 200 ± 2 grams on the specimen. Its contacting surface and the surface of the anvil shall be parallel within 0.0001 inch.

4. Procedure

The specimen shall be placed on the anvil, and the presser foot lowered gently (not dropped) until it rests on the specimen. The thickness shall be read from the dial to 0.001 inch.

The number and location of the thickness measurements taken shall be as directed in the particular physical test method being used.

5. Results

The thickness of the sample shall be reported with the results obtained from the test in which the specimen is used.

6. History and Status

The method was part of a method for "Measuring the Dimensions of Leather" adopted as official in 1941.

WIDTH (P4)

1. Scope

This method is intended for use in determining the width of physical test specimens and of regularly-shaped units and pieces of all types of leather.

2. Specimen

The specimen shall be the prepared test specimen, unit, or piece.

3. Apparatus

THE APPARATUS SHALL CONSIST OF

A. A steel scale graduated to 0.02 inch for measuring the width of test specimens and small regular pieces.

B. A steel tape graduated to $^1/_{16}$ inch for measuring large units and pieces.

4. Procedure

Test specimens. The specimen shall be laid on a flat surface and flattened out without pulling or stretching. The width shall be measured to the nearest 0.02 inch at the locations designated in the method for making the particular test for which the specimen was prepared.

Small pieces. The piece shall be laid on a flat surface and flattened out without pulling or stretching. The width shall be measured to the nearest 0.02 inch in at least three places equally spaced along the specimen.

Large units and pieces. The unit or piece shall be laid on a flat surface and flattened as well as possible without pulling or stretching. The width shall be measured in at least 3 places equally spaced along the specimen. The measurements shall be taken to the nearest $^1/_{16}$ inch.

5. Results

The width of test specimens shall be used or reported as designated in the method for making the particular physical test for which the specimen was prepared.

The average of the width measurements taken on any unit or piece shall be the width of the piece. The average shall be reported to the same accuracy as the measurements were taken.

6. History and Status

This method was part of a method for measuring the "Dimensions of Leather" which was adopted as official in 1941.

TONGUE TEAR STRENGTH (P10)

Tongue Tear Strength is defined as the load required to tear the leather between two tongues formed by splitting the leather perpendicular to its surface.

1. Scope

This method is intended for use on all types of leather.

2. Specimen

The specimen shall be cut to the dimensions shown in Figure 1.

It shall be a rectangular piece of leather, 1 inch by 4 inches by the full thickness of the unit. It shall have a $^3/_{16}$ inch diameter hole located on the long axis 1 inch from one end and shall be split along its axis from the hole

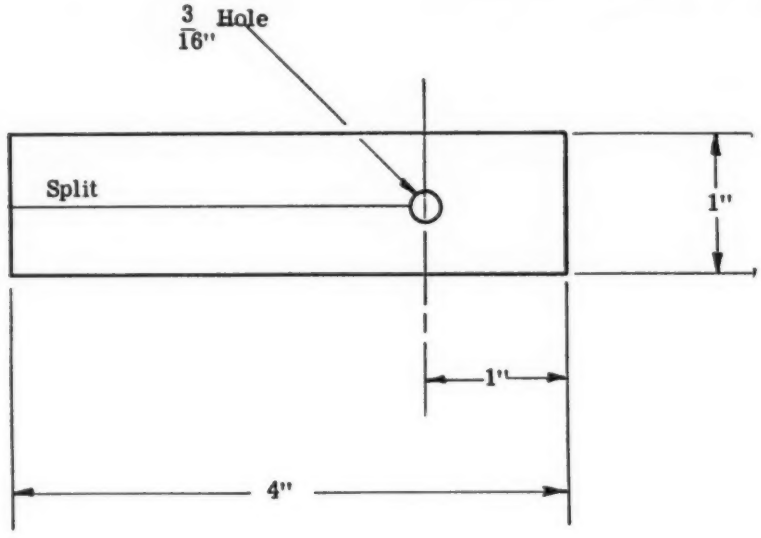


FIG. 1

to the other end to form two tongues. The direction of the long axis of the specimen relative to the backbone shall be noted.

3. Apparatus

THE APPARATUS SHALL CONSIST OF

A. A testing machine as described in the method (P15) for "Tensile Strength".

B. A spring-type thickness gage, as described in the method (P2) for measuring the "Thickness of Units".

C. A $^{3}/_{16}$ inch punch.

D. A knife for cutting and splitting the specimen.

E. A steel scale graduated to 0.02 inch.

4. Procedure

The thickness of the specimen shall be determined to the nearest 0.5 ounce on the long axis between the hole and the unsplit end of the specimen. The gage foot shall be adjacent to, but shall not overlap, the hole.

One of the tongues on the specimen shall be clamped in each of the testing machine grips so that the long axis of the tongue is in the direction of the

motion of the movable grip. The faces of the grips shall be approximately 2 inches from the common base of the tongues. The machine shall be operated at 10 ± 2 inches per minute until the specimen starts to tear. At the instant of the initial tear, the load registered by the machine shall be noted and recorded.

5. Results

The thickness measurements of all the specimens tested shall be averaged to the nearest 0.5 ounce and reported as the thickness of the sample.

The loads required to tear the specimens shall be averaged to the nearest pound and reported as the Tongue Tear Strength of the sample.

The direction of the long axis of the specimen relative to the backbone shall be reported.

6. History and Status

This method was part of a method for measuring the "Tearing Strength" of leather which was adopted as official in 1947.

BUCKLE TEAR STRENGTH (P11)

Buckle Tear Strength is defined as the load required to tear a leather strap fastened in a buckle.

1. Scope

This method is intended for use on all types of leather.

2. Specimen

The specimen shall be a piece of leather 1 inch by 8 inches. The direction of the long axis relative to the backbone shall be noted.

3. Apparatus

The Apparatus Shall Consist of

A. A spring-type thickness gage as described in the method (P2) for measuring the "Thickness of Units".

B. A steel punch which will make a 3/16 inch diameter hole.

C. A 1 1/4 inch harness buckle with a 0.175 ± 0.005 inch diameter tongue. It shall be attached to a strong strap, 1 1/4 inch wide and at least 5 inches long. The strap shall be equipped with a keeper located approximately 3/4 inch from the buckle.

D. A testing machine as described in the method (P15) for "Tensile Strength".

4. Procedure

A $^{5}/_{16}$ inch hole shall be punched on the long axis of the specimen 3 inches from one end.

The thickness of the specimen shall be measured to the nearest 0.5 ounce on the long axis between the hole and the short end. The gage foot shall be adjacent to, but shall not overlap, the hole.

The specimen shall be fitted in the buckle with the tongue of the buckle in the hole and the short end of the specimen under the keeper. The other end of the specimen shall be clamped in one of the testing machine grips so that the hole is approximately 3 inches from the grip. The free end of the buckle strap shall be clamped in the other grip. The machine shall be operated at 10 ± 2 inches per minute until the specimen begins to tear. At that instant the load registered by the machine shall be noted and recorded.

5. Results

The thickness of the sample shall be the average of the thickness measurements of the specimens and shall be reported to the nearest 0.5 ounce.

The Buckle Tear Strength of the sample shall be the average of the loads for the specimens and shall be reported to the nearest pound.

The direction of the long axis relative to the backbone shall be reported.

6. History and Status

This method was part of a method for measuring the tearing strength of leather which was adopted as official in 1947.

STITCH TEAR STRENGTH, SINGLE HOLE (P12)

The Single Hole Stitch Tear Strength is defined as the load required to tear a leather specimen from a rod through a hole in the specimen.

1. Scope

This method is intended for use on heavy leathers.

2. Specimen

The specimen shall be a piece of leather, 1 inch by 2 inches. The end of the specimen in which the hole is to be punched shall be at least 1 inch away from any uncut edge on the unit. The direction of the long axis relative to the backbone shall be noted.

3. Apparatus

The Apparatus Shall Consist of

A. A spring-type thickness gage as described in the method (P2) for measuring the "Thickness of Units".

B. A steel punch, beveled on the outside, which will make a 1/8 inch diameter hole.

C. A metal yoke made to the dimensions shown in Figure 1.

D. A steel rod 0.095 ± 0.005 inch in diameter and at least 1 inch long.

E. A testing machine as described in the method (P15) for "Tensile Strength". The jaws of one of the grips shall be covered to protect them from possible damage caused in clamping the metal yoke.

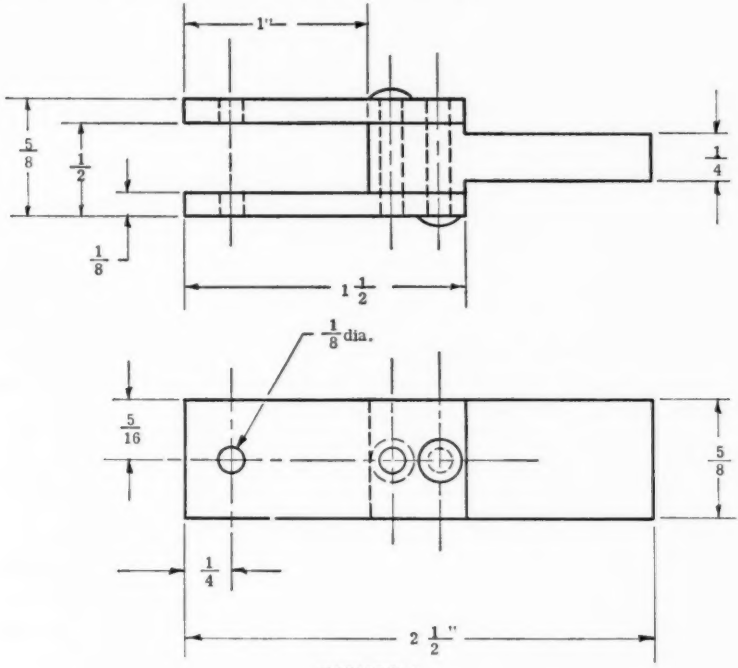


FIGURE 1.

4. Procedure

The thickness of the specimen shall be determined to the nearest 0.5 iron on the long axis of the specimen near the end in which the hole is to be punched.

A 1/8 inch diameter hole shall be punched on the long axis of the specimen the center of the hole being 5/32 inch from the end on which the thickness was measured.

The yoke shall be gripped in one of the testing machine jaws. The end of the specimen containing the hole shall be placed between the arms of the yoke, and the steel rod shall be passed through the holes in the yoke and the

specimen. The free end of the specimen shall be clamped in the other testing machine grip. The machine shall be operated at 10 ± 2 inches per minute until the specimen starts to tear. At the instant that tearing begins, the load registered by the machine shall be noted and recorded.

5. Results

The thickness measurements of all the specimens tested shall be averaged to the nearest 0.5 iron and reported as the thickness of the sample.

The loads required to tear the specimens shall be averaged to the nearest pound and reported as the Single Hole Stitch Tear Strength of the sample.

The direction of the long axis of the specimen relative to the backbone shall be reported.

6. History and Status

This method was part of a method for measuring the "Tearing Strength" of leather which was adopted as official in 1947.

STITCH TEAR STRENGTH, DOUBLE HOLE (P13)

Double Hole Stitch Tear Strength is defined as the load required to tear the leather between two holes a given distance apart.

1. Scope

This method is intended for use on light leathers.

2. Specimen

The specimen shall be a piece of leather 1 inch by 2 inches. The end of the specimen in which the holes are to be punched shall be at least 1 inch away from any uncut edge on the unit. The direction of the long dimension relative to the backbone shall be noted.

3. Apparatus

The Apparatus Shall Consist of

A. A spring-type thickness gage as described in the method (P2) for "Thickness of Units".

B. A steel punch which will make a hole $^{5}/_{64}$ inch diameter.

C. A soft steel wire 0.040 ± 0.001 inch in diameter and not less than 2 $^{1}/_{4}$ inches long. The large loop of a No. 1 paper clip meets the requirements.

D. A testing machine as described in the method (P15) for "Tensile Strength". The jaws of one of the grips shall be covered to protect them from possible damage caused in clamping the metal wire.

4. Procedure

The thickness of the specimen shall be measured to the nearest 0.5 ounce on the long axis near one end.

Two $^{5}/_{64}$ inch diameter holes shall be punched in the end of the specimen on which the thickness was measured. The holes shall be located as Shown in Figure 1.

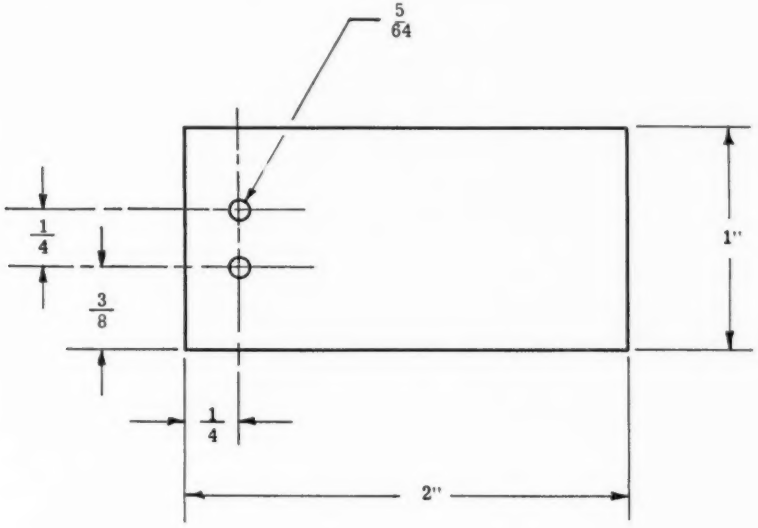


FIG. 1

The wire shall be bent into a "U" shape, preferably by bending over a $^{1}/_{4}$ inch rod, and the ends passed through the holes in the specimen so that both ends project from the flesh side of the specimen. Both ends of the wire shall be clamped in the testing machine grip, the jaws of which have been covered. The free end of the specimen shall be clamped in the other grip. The machine shall be operated at 10 ± 2 inches per minute until the specimen tears. At the instant that the specimen begins to tear, the load registered by the machine shall be noted and recorded.

5. Results

The thickness measurements of all the specimens tested shall be averaged to the nearest 0.5 ounce and reported as the thickness of the sample.

The loads required to tear the specimens shall be averaged to the nearest pound and reported as the Double Hole Stitch Tear Strength of the sample.

The direction of the long axis of the specimen relative to the backbone shall be reported.

6. History and Status

This method was part of a method for measuring the tearing strength of leather which was adopted as official in 1947.

BURSTING STRENGTH, BALL (P14)

Ball Bursting Strength is defined as the load in pounds required to force a ball-ended plunger through a leather diaphragm.

1. Scope

This method may be used to test all light leathers and leather products.

2. Specimen

The specimen shall be a disc of leather at least 1 3/4 inches in diameter. (When a number of adjacent tests are desired, a strip of leather between 1 3/4 and 6 inches wide may be used for a specimen. When tests are made on such a strip, the clamping areas can be overlapped so that tests can be made with a minimum distance of 1 1/4 inches between the bursts.)

3. Apparatus

The Apparatus Shall Consist of

A. A thickness gage, as described in the method (P3), "Thickness of Specimens".

B. A specimen holder, as illustrated in Figure 2 and Figure 3, or equivalent.

C. A testing machine, as described in the method (P15), "Tensile Strength", except that the machine shall be arranged to have a cross head speed of 3 inches ± 1 inch per minute.

4. Procedure

The specimen shall be cut with a die, knife, or shears. Its thickness shall be measured to the nearest 0.001 inch in at least 3 places near its center. (If a strip is used as a specimen, the centers of all the test areas shall be located and properly marked, and the thickness at each center shall be determined before any tests are made on the strip.)

The specimen holder shall be mounted in the machine. The specimen shall be placed in the holder, grain up. The space between gripping surface adjusted in accord with the thickness of the specimen, and the specimen gripped

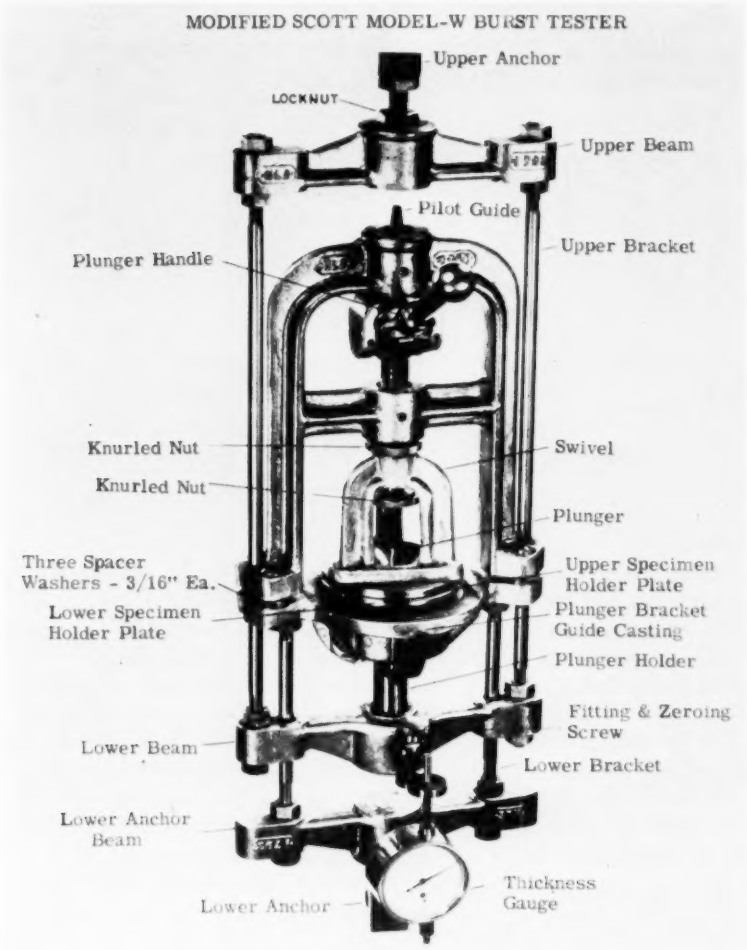


FIGURE 1.

securely by pulling the plunger handle downward. The machine shall be operated at 3 inches ± 1 inch per minute. If the grain crack load is desired, the specimen shall be observed for grain cracking. At the instant the grain cracks, the load registered by the machine shall be noted without stopping the machine, and recorded as the grain cracking load. The test shall continue until the specimen bursts. The maximum load registered by the machine shall be recorded as the bursting strength.

PARTS REQUIRED TO MODIFY MODEL-W SCOTT BURST TESTER

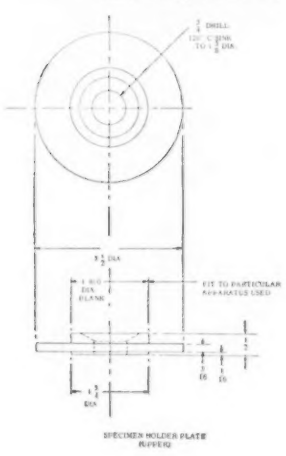


FIG. 2

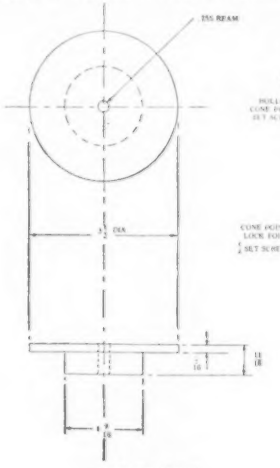


FIG. 3.

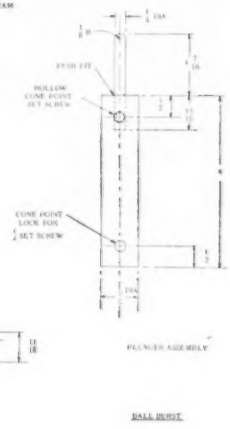


FIG. 4

5. Results

The thickness measurements taken on a specimen shall be averaged to the nearest 0.001 inch to obtain the thickness of the specimen.

The thickness measurements of all the specimens tested shall be averaged to the nearest 0.001 inch and recorded as the thickness of the sample.

The grain crack loads for all the specimens tested shall be averaged to the nearest pound and reported as the "Grain Crack Load" of the sample.

The bursting loads for all the specimens tested shall be averaged to the nearest pound and reported as the "Bursting Load" of the sample.

The bursting load for the sample may be calculated as pounds per inch of thickness by dividing the average bursting load by the average thickness of the specimens.

6. History and Status

A method for "Bursting Strength" in which the Mullen Tester was used was adopted as official in 1948.

TENSILE STRENGTH (P15)

Tensile Strength is defined as the load required to rupture a test specimen under tension divided by the area of the original cross section of the specimen.

1. Scope

This method is intended for use on all types of leather except leathers which are not smooth and firm enough to permit accurate thickness measurements.

2. Specimens

Test specimens shall be cut with a die of the shape shown in Figure 1. The direction of the axis of the specimen relative to the backbone shall be noted.

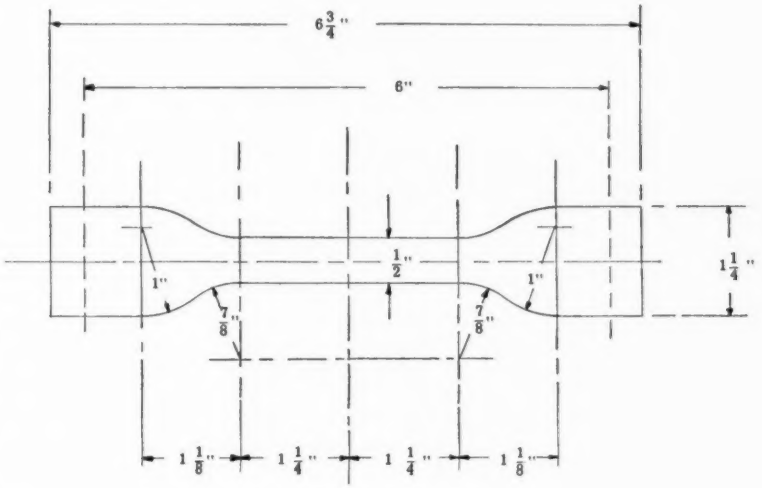


FIG. 1

3. Apparatus

THE APPARATUS SHALL CONSIST OF

A. A dead-weight type of thickness gage as described under the method (P3) for measuring the "Thickness of Specimens".

B. A steel scale graduated to read in 0.02 inch.

C. A steel die designed to cut specimens either 6 inches or 6 3/4 inches long and of the other dimensions shown in Figure 1. The cutting edges shall be beveled on the outside and straight on the inside. The die shall be kept sharp and free from nicks to give clean, even edges on the specimen. (A 6-inch specimen may be used when the longer die is not available.)

D. The cutting block shall consist of a flat, hardwood support, upon which is placed a flat, smooth sheet of firm material that will not injure the cutting edge of the die. For this purpose, the following materials are suggested: Rubber belting, leather belting, light cardboard, or 13 gage aluminum.

E. The testing machine shall be power-driven and the applied force shall be indicated on a dial, scale, or chart. The load indicator shall remain at the point of maximum load after the specimen breaks. The speed of the power-actuated grip shall be 10 ± 2 inches per minute when running free and shall

be uniform at all times. The error of the machine up to a load of 50 pounds shall not exceed 2 per cent; at loads of more than 50 pounds the errors shall not exceed 1 per cent.

F. The machine shall be equipped with a set of grips for clamping the specimens. The faces of the grips shall be knurled or otherwise roughened to prevent slipping of the specimen. The gripping surfaces shall be 1 inch by 1½ inches. The grips shall be mounted with the longer dimension perpendicular to the direction of the application of the load.

4. Procedure

The thickness of the specimen shall be measured to the nearest 0.001 inch at three points: At the mid-point of the narrow section and at a distance 0.75 inch each side.

The width of the specimen shall be measured to the nearest 0.02 inch at three places: At the mid-point of the narrow section and at a distance 0.75 inch each side.

The specimen shall be mounted symmetrically with the jaws when they are separated such a distance that the ends of the specimens, when clamped, shall extend to or slightly beyond the back edge of the jaws.

The machine shall be started and when the specimen has been ruptured, the load in pounds shall be recorded.

5. Results

The original cross-sectional area of the specimen shall be calculated using the median thickness and width measurements.

The tensile strength shall be calculated to the nearest 10 pounds per square inch by dividing the load in pounds by the area in square inches of the original cross section of the narrow portion of the specimen.

The report shall state the direction of the long axis of the specimen relative to the backbone.

6. History and Status

A method for measuring tensile strength of heavy leather, narrow leather, and soft leather was adopted as official by the Association in 1943.

BREAKING STRENGTH (P16)

Breaking strength is defined as the load in pounds required to rupture a specimen of leather.

1. Scope

This method is intended for use on light, soft leathers; boarded, buffed, or embossed leathers; narrow strap, welt, lace, and round belt leathers, or other leathers which cannot be tested by the prescribed Tensile Strength Method.

2. Specimens

The specimen for wide, flat leathers shall be a piece of leather 6 ± 1/16 inches long and 4 ± 1/16 inches wide. The direction of the long dimension relative to the backbone shall be noted.

The specimen for narrow strap, welting, lace, and round belt leathers shall be a piece of leather 6 ± 1/16 inches long.

3. Apparatus

The Apparatus Shall Consist of

A. A testing machine as described in the method (P15) for "Tensile Strength", except that the face of one jaw of each clamp shall measure 1 inch by 1 inch and that of the other shall measure 1 inch by 1½ inches or more.

B. A spring-loaded thickness gage as described in the method (P2) for measuring the "Thickness of Units".

C. A steel scale graduated to 0.02 inch.

4. Procedure

For Testing Wide Specimens (Grab Method).

The thickness of the specimen shall be measured to the nearest 0.5 ounce in three places—at the center, and on the long axis one inch each side of the center.

The distance between the grips at the start of the test shall be 3 ± 1/8 inches.

The specimen shall be placed in the testing machine, with its long axis parallel to the direction of the applied force, and clamped lightly. The material of the specimen shall be distributed uniformly and made to hang flat between the jaws before the specimen is tightly clamped.

The machine shall be operated so that the movable clamp travels at a speed of 10 ± 2 inches per minute.

The load required to rupture the specimen shall be recorded as the breaking strength. (In a test of this type, many of the specimens will rupture at the jaws of the testing machines. The load should be recorded regardless of the place of rupture.

For Testing Narrow Specimens.

The thickness of a flat specimen or the diameter of a round specimen shall be measured to the nearest 0.5 ounce in three places—at the center, and 1 inch each side of center.

The width of flat specimens shall be measured to the nearest 0.02 inch at the same three places.

The specimen shall be placed in the machine with its long axis parallel to the direction of the applied force.

The machine shall be run so that the movable clamp travels at 10 ± 2 inches per minute.

The load required to rupture the specimen shall be recorded as the breaking strength.

5. Results

The average of the thickness measurements made on all specimens shall be the thickness of the sample and shall be reported to the nearest 0.5 ounce.

The average of the width measurements made on all the specimens shall be the width of the sample and shall be reported to the nearest 0.02 inch.

The average of the breaking strengths obtained from the specimens tested shall be the breaking strength of the sample and shall be reported to the nearest pound.

6. History and Status

This method was part of the method for "Measuring the Tensile Strength of Leather" and was adopted as official in 1943.

ELONGATION (P17)

Elongation (or stretch) is defined as the increase in length of specimen under tension. It is generally measured at a designated load and expressed as a percentage of the original length.

1. Scope

This method is intended for use on all types of leathers.

2. Specimen

When the unit from which the specimen is to be cut is of sufficient size, the specimen shall be cut with the die used in preparing specimens for tensile strength tests. The direction of the long axis relative to the backbone shall be noted. (Elongation cannot be determined on the type of specimen used in obtaining the breaking strength by the grab method. The elongation of soft leathers and boarded, buffed, or embossed leathers can be accurately determined on a specimen of the shape used in testing for tensile strength).

The specimen for narrow strap, welting, lace, and round belt leathers shall be a piece of leather 6 ± $1/16$ inches long.

3. Apparatus

The Apparatus Shall Consist of

A. A testing machine as described under the method (P15) for "Tensile Strength".

B. A bench marker, such as is shown in Figure 1, having a frame on which two parallel knives with marking edges are mounted.

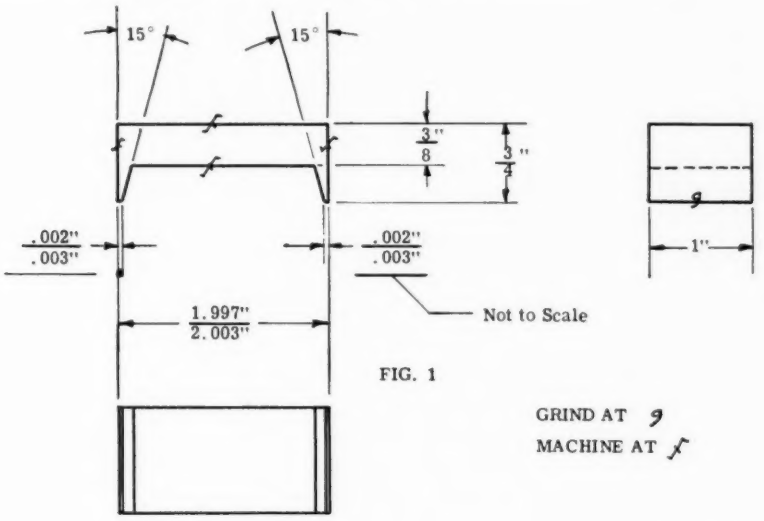


FIG. 1

The marking edges shall be 1 inch long and 0.002 to 0.003 inch wide and shall be smooth, true and free of burrs. The outer sides of the knives shall be perpendicular with the marking surface, the inner sides shall be at an angle of 15° with the perpendicular. The distance between the centers of the marking edges shall be 2 ± 0.003 inches.

C. A stamp pad having a plane firm surface of either hardwood, glass or plastic on which a film of ink can be spread.

D. Ink of such quality that it will clearly mark the specimen without affecting the strength or stretch of the leather.

E. Dividers having at least 4 inch legs and which are screw-adjustable.

F. Thickness gages as follows.

For procedure A, a dead-weight gage as described in the method (P3) for measuring the "Thickness of Specimens".

For procedure B, a spring-type gage as described in the method (P2) for measuring the "Thickness of Units".

G. A steel scale graduated to read in 0.02 inch.

4. Procedure

Procedure A. When either the tensile strength or the breaking strength and the elongation are to be determined, both values shall be obtained on the same specimen at the same time. All the requirements of the strength

test and the following extra procedures necessary for obtaining the elongation shall be met.

A. The specimen shall be marked, using the inked bench marker, with two light but legible parallel lines across and perpendicular to its long axis and equal distances each side of its center.

B. The specimen shall be mounted as directed in the strength test and the machine shall be set in motion at 10 ± 2 inches per minute.

C. The distance between the bench lines on the specimen shall be followed with the dividers until the load at which the elongation is desired has been reached. The distance between the points of the dividers shall be measured to the nearest 0.02 inch.

D. The machine shall not be stopped when the load at which the elongation is to be measured has been reached but shall continue until the specimen breaks.

Procedure B. When the elongation is to be determined separately, the following procedure shall be followed.

A. The thickness of the specimen shall be determined at its center.

B. The specimen shall be marked as described in Procedure A.

C. If the dumbbell specimen is to be used, it shall be mounted as described in the method (P15) for "Tensile Strength". If a narrow specimen is to be used, it shall be mounted as described in the method (P16) for "Breaking Strength". The machine shall be set in motion at 10 ± 2 inches per minute and run until the load at which the elongation is desired has been reached.

D. The elongation shall be measured as described in Procedure A.

5. Results

The elongation shall be calculated to the nearest 0.1 per cent of the original length between the bench marks.

The percentage elongation of all the specimens tested shall be averaged to obtain the elongation of the sample.

6. History and Status

The method for measuring elongation was adopted as provisional in 1943, revised in 1945, made official in 1947.

ALTERNATE METHOD FOR ELONGATION (P17A)

The following method is not an official method of the Association but may be used to obtain comparative elongation values, provided similar specimens are tested under similar conditions.

1. Scope

This method can be used to measure the elongation of all types of leathers.

2. Specimen

When the unit from which the specimen is to be cut is of sufficient size, the specimen shall be cut with the die used in preparing specimens for tensile strength tests. The direction of the long axis relative to the backbone shall be noted. (Elongation cannot be determined on the type of specimen used in obtaining the breaking strength by the grab method. The elongation of soft leathers and boarded, buffed, or embossed leathers can be accurately determined on a specimen of the shape used in testing for tensile strength.)

The specimen for narrow strap, welting, lace, and round belt leathers shall be a piece of leather $6 \pm {}^{1}/_{16}$ inches long.

3. Apparatus

The Apparatus Shall Consist of

A. A testing machine as described under (P15) "Tensile Strength", except that in addition to other requirements it shall have an attachment for automatically and simultaneously recording both load and relative displacement of the jaws. (An autographic Scott Tester and other machines with autographic recording devices meet these requirements.)

B. Thickness gages as follows.

For procedure A, a dead-weight gage as described in the method (P3) for measuring the "Thickness of Specimens".

For procedure B, a spring-type gage as described in the method (P2) for measuring the "Thickness of Units".

C. A steel scale graduated to 0.02 inch.

4. Procedure

Procedure A. When either the tensile strength or breaking strength and the elongation are to be determined, both values shall be obtained on the same specimen at the same time. All the requirements of the strength test and the following extra procedures necessary for obtaining elongation shall be met.

After the specimen is mounted in the jaw, the machine shall be carefully run until the specimen just becomes taut. The machine shall be stopped and the distance between the jaws shall be measured to the nearest 0.02 inch.

The autographic attachment shall be set to zero.

The machine shall be set in motion at 10 ± 2 inches per minute and run until the specimen ruptures.

The elongation at the desired load shall be read from the chart and shall be calculated as a percentage of the original distance between the jaws.

Procedure B. When the elongation is to be determined separately, the following procedure shall be followed.

The thickness of the specimen shall be determined at its center.

If the dumbbell specimen is to be used, the specimen shall be mounted as described in the method (P15) for "Tensile Strength". If a narrow specimen is to be used, the specimen shall be mounted as described in the method (P16) for "Breaking Strength". The distance between the jaws shall be determined as described in Procedure A.

The machine shall be set in motion at 10 ± 2 inches per minute and run until the load at which the elongation is desired has been reached. The elongation, at the desired load, shall be read from the chart and shall be calculated as a percentage of the original distance between the jaws.

5. Results

The elongation shall be calculated to the nearest 0.1 per cent of the original distance between the jaws.

The percentage elongation of all the specimens tested shall be averaged to obtain the elongation of the sample.

6. History and Status

A method for measuring the "Elongation" of leather was adopted as provisional in 1943, revised in 1945, and made official in 1947.

WATER ABSORPTION (Static) (P30)

Water Absorption (Static) is defined as the amount of water absorbed by leather immersed, without flexing, in water for a given time.

1. Scope

This method is intended for use on all types of leather.

2. Specimen

The specimen shall be a piece of leather approximately 2 by 4 inches.

3. Apparatus

The Apparatus Shall Consist of

A. An immersion dish of glass or other non-corrosive material, having a flat bottom at least 3 by 5 inches.

B. A balance sensitive to 0.01 gram.

C. A drying oven with automatic temperature control.

D. A spring-type thickness gage as described in the method (P2) for "Thickness of Units".

4. Procedure

The conditioned specimen shall be lightly brushed or blown to remove any loose adhering material. Its thickness shall be measured in at least 3 places

to the nearest 0.5 ounce or 0.5 iron, depending on the type of leather. The specimen shall be weighed to the nearest 0.01 gram and its weight recorded as (W1).

The bottom of the immersion dish shall be sparsely covered with glass beads. The specimen with the grain side up shall be placed flat on the beads and weighted with pieces of glass rod to keep it immersed. Distilled water at 23° C. ± 3° C. shall be carefully poured into the dish until the specimen is fully immersed, at least 10 grams of water per gram of specimen being used. Air bubbles under the specimen shall be removed by carefully tilting the dish. Flexing of the specimen shall be avoided.

Two immersion periods, one of 30 minutes and one of 2 hours, shall be used. (The use of a 24-hour immersion period is optional.)

A. After the water is added, the specimen shall remain immersed for 30 minutes. It shall be taken from the dish, the surface water shall be removed with blotting paper, and the specimen shall be weighed immediately to the nearest 0.01 gram. The weight shall be recorded as ($W2_1$).

B. The specimen shall remain in the air for a total of 5 minutes, including the weighing time, when it shall be reimmersed and allowed to remain immersed for a total of 2 hours including the original 30-minute immersion period, giving a total testing time of 2 hours and 5 minutes. The specimen shall be removed, blotted, and weighed. The weight shall be recorded as ($W2_2$).

If a 24-hour immersion period is used, the specimen shall be reimmersed 5 minutes after being removed from the water and allowed to soak for 24 hours including the 2-hour immersion period. It shall then be removed from the immersion dish, blotted, and weighed. The weight shall be recoreded as ($W2_3$).

When correction for the water solubles removed during immersion is desired, it shall be made as follows. At the end of any immersion period, the water in the immersion dish shall be thoroughly stirred so that all extracted material is in suspension or solution, transferred to a beaker, and weighed to the nearest 0.1 gram. The total weight of the water shall be recorded as (Ww). An aliquot of 25 milliliters of the water shall be taken from the beaker, put in a weighed tannin dish, and dried at 105° C. The tannin dish and residue shall be weighed and the weight (Wr) of the residue determined. The total weight of the extracted water solubles shall be computed as

$$W3 = \frac{Ww \times Wr}{25}$$

The remaining water shall be returned to the immersion dish and the test continued.

5. Results

When correction for extracted water solubles is not made, the water absorption of the specimen shall be calculated to the nearest 0.1 per cent as follows.

$$A = \frac{W2 - W1 \times 100}{W1}$$

where

A = Water Absorption = per cent water absorbed
W1 = original weight of the specimen
W2 = wet weight of specimen after any period of immersion

When correction for extracted water solubles is made, the water absorption shall be calculated to the nearest 0.1 per cent as follows.

$$A = \frac{W2 + W3 - W1 \times 100}{W1}$$

where

A, W1, W2 have significance as shown above.
W3 = weight of the extracted water solubles.

The water absorption of the sample shall be the average of the percentages of water absorbed by the specimens. The report shall state the time of immersion.

The thickness measurements of all the specimens tested shall be averaged to the nearest 0.5 ounce or 0.5 iron and reported as the thickness of the sample.

6. History and Status

A method for determining the water absorption of sole leather was adopted as provisional by ALCA in 1943. It was changed and continued as provisional in 1947.

WATER RESISTANCE (Dynamic) (P31)

Water Resistance (Dynamic) comprises the following three factors.

A. Water Penetration, which is measured by the number of times a leather specimen, having one side in contact with water, must be flexed before water penetrates through the leather.

B. Water Transmission, which is the amount of water which passes through the wet specimen during a given number of flexing cycles.

C. Water Absorption, which is the amount of water retained by the specimen after the penetration and transmission measurements.

1. Scope

This method is intended for use on all flexible leather 1/8 inch or less in thickness. It cannot be used on stiff leathers such as sole and belting leathers.

2. Specimen

The specimen shall be a rectangular piece of leather between 3 3/4 and 3 7/8 inches wide and between 4 1/4 and 4 3/8 inches long.

3. Apparatus

The Apparatus Shall Consist of

A. A balance sensitive to 0.01 gram.

B. A water penetration machine as shown in Figure 1 or its equivalent. (Complete working drawings for making this machine can be secured from the Secretary of the ALCA.)

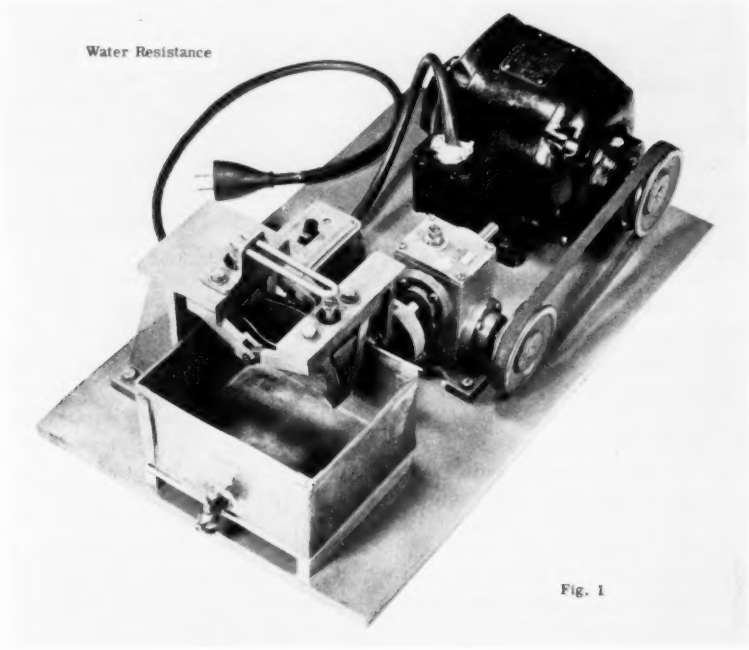


Fig. 1

It Shall Have the Following Elements

A specimen holder made of two V-shaped clamps with wedges for holding the specimen. The clamps shall be 2.5 ± 0.1 inches apart, inside measure-

ment, when their tops are in the same horizontal plane. One clamp shall be fixed to the frame. The other clamp shall be pivoted as shown in Figure 1 and attached, through a connecting link, to a motor-driven eccentric which turns at 90 RPM. In one rotation of the eccentric the clamp shall move a distance of 1 ± 0.05 inch below the horizontal and return.

A water tank made of copper, stainless steel, or other non-corrosive material. It shall be of such a size and depth that it can be placed around the clamps and filled with water to such a level that, when the tops of the clamps are in the same horizontal plane, the lowest point of each clamp is 1.00 ± 0.05 inch below the surface of the water.

A base for the water tank which is removable from between the machine frame and the water tank.

A counter, preferably a reset counter, connected to the movable clamp to record the number of cycles through which the specimen is flexed.

C. A 2 x 4 inch wick made by folding a piece of chamois into at least 4 layers. The chamois shall meet the requirements of Federal Specifications KK-S-416.

D. A spring-type thickness gage as described in the method (P2) for "Thickness of Units".

4. Procedure

Water Penetration, Water Transmission and Water Absorption shall be determined on the same specimen at the same time.

The thickness of the specimen shall be measured to the nearest 0.5 ounce in at least three places near the center of the specimen.

The weight of the specimen shall be determined to the nearest 0.01 gram.

Immediately prior to use, the wick shall be wet thoroughly, wrung out, and weighed to the nearest 0.01 gram.

The machine shall be set so that the tops of both clamps are in the same horizontal plane. The specimen with the grain side out shall be fastened in the clamps by means of the V-shaped wedges so that a trough is formed with the leather sides under slight tension. The water tank shall be put in place and filled with distilled water to a level 1.00 ± 0.05 inch above the lowest point of the clamps. The counter-reading shall be recorded.

Water Penetration. The movable clamp shall be set in operation and run until water appears inside the leather. (The water usually first appears as small beads which are easily seen. If beads are not formed, very careful observation is necessary to determine initial penetration.) At the moment that water appears, the machine shall be stopped and the counter reading recorded.

Water Transmission. The weighed chamois wick shall be placed in the trough in such a manner that it will absorb any water which penetrates the

specimen. Care must be used in placing the wick not to upset or change the fold in the leather across the bottom of the trough. The apparatus shall be set in motion and run the desired number of cycles, X_2 (usually 1350). The machine shall then be stopped and the wick immediately weighed to the nearest 0.01 gram.

Water Absorption. The water shall be drained and the tank removed without splashing water into the trough. The specimen shall be removed from the machine, blotted with absorbent paper, and weighed to the nearest 0.01 gram.

5. Results

The thickness of the specimen shall be the average of the thickness measurements. It shall be calculated to the nearest 0.5 ounce. The average of the thickness measurements of all the specimens shall be the thickness of the sample. It shall be calculated to the nearest 0.5 ounce and reported.

The original counter reading shall be subtacted from the counter reading at the time water penetrated the specimen, and the difference (X_1 cycles) reported as Water Penetration.

The original weight of the wrung-out wick shall be subtracted from its final weight, and the difference reported as Water Transmission in X_2 cycles.

The original weight of the specimen shall be subtracted from its final weight and the result reported as Water Absorption in $X_1 + X_2$ cycles.

PERMEABILITY TO WATER VAPOR (P32)

Permeability to Water Vapor is defined as the rate at which water vapor passes through a material exposed to moist air on one side and dry air on the other.

1. Scope

This method is intended for use on all types of leathers.

2. Specimen

The specimen shall be a disc of leather, 2.75 ± 0.1 inches in diameter.

3. Apparatus and Reagents

The Apparatus and Reagents Shall Consist of

A. An analytical balance accurate to 0.01 gram.

B. A permeability apparatus as shown assembled in Figure 1.

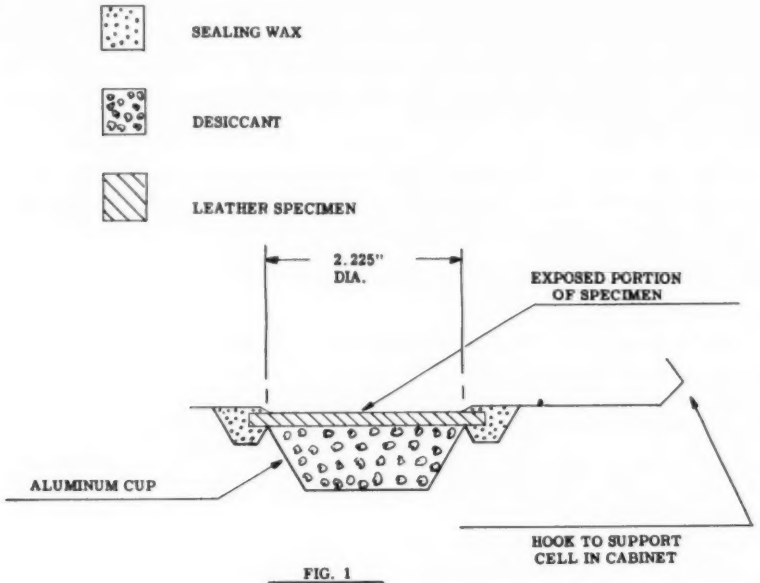


FIG. 1

It shall consist of an aluminum cup having approximately the dimensions shown in Figure 2 and a template, $^1/_8$ inch thick and 2.225 ± 0.025 inches in diameter (area 25 sq. cm.), made of brass or other non-corrosive metal.

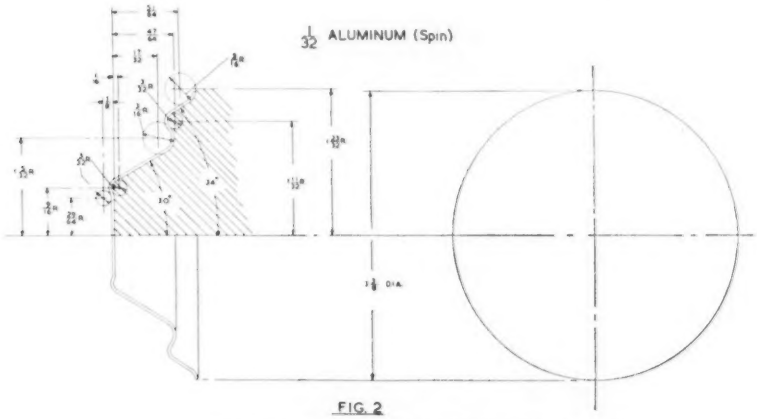


FIG. 2

SPINNING STUD FOR WATER VAPOR PERMEABILITY DISH

C. A steel stamping die, 2.75 ± 0.1 inches in diameter.

D. A constant temperature and humidity chamber which may be either a laboratory, a cabinet, or a chamber with circulated air maintained at standard condition (23° C ± 1° C. and 50± 4 per cent relative humidity).

E. A desiccant which shall be 8 mesh anhydrous calcium chloride.

F. Microcrystalline wax with a melting point of 70° C. or lower.

4. Procedure

The aluminum cup shall be filled with the desiccant to the level of the rim, as shown in Figure 1. The specimen, with the grain side downward, shall be placed over the desiccant and centered with the cup. The template shall be placed flat on the specimen and centered as nearly as possible with the cup. A slight pressure shall be applied to the template to insure good contact between the rim of the cup, the specimen, and the template around the entire rim. The groove formed by the lip of the cup and the edge of the template shall be filled with melted wax which is allowed to harden before the template is removed. (The removal of the template from the hardened wax is facilitated by applying a coat of petroleum jelly to the edge and chilling the template prior to use.)

The complete assembly shall be placed in the constant temperature and humidity chamber. At the end of one hour, the assembly shall be weighed to the nearest 0.01 gram. (The assembly shall preferably be weighed in the constant temperature and humidity chamber. If this is not possible, the assemblies can be removed and weighed one at a time, provided they are returned to the chamber in two minutes or less.) The assembly shall be weighed at 0.5 hour intervals until three or more successive weighings show weight increases which are equal within 0.01 gram. (The weighing intervals can be extended to one hour or more for specimens with low permeability.)

5. Results

The permeability to water vapor of the specimen shall be calculated by the formula

$$P = \frac{I}{AT}$$

where

P = water vapor permeability in grams per square centimeter per hour
I = increase in weight in grams in 0.5 hour
A = area of the exposed specimen in square centimeters (normally 25)
T = time of exposure in hours

The average of the results obtained from the specimens tested shall be the Permeability to Water Vapor of the sample.

6. History and Status

A method, in which a tannin dish was filled with activated aluminum oxide and covered with a disc of leather placed between two brass rings with center openings 1 inch in diameter, was made official in 1941.

PIPING (P40)

Piping is defined as the formation of coarse wrinkles in the grain layer transverse to the direction in which a leather specimen is bent. Small uniform wrinkles which are associated with the break of firm leathers are not to be considered as piping.

1. Scope

This method is intended for use on all types and thicknesses of leather.

2. Specimen

The specimen shall preferably be a piece of leather, 1 inch wide and at least 4 inches long, cut with its long edges at least 1 inch away from any uncut edge of the unit. To save cutting a specimen from a unit of light leather, a 2-inch width of the edge can be tested. The direction of the long axis of the specimen relative to the backbone shall be recorded.

3. Apparatus

The Apparatus Shall Consist of

A. Thickness gages of the wedge or spring-types as described in the method (P2) for measuring the "Thickness of Units".

B. A set of mandrels consisting of eleven metal pins at least 4 inches long with diameters of 1.688, 1.063, 0.750, 0.563, 0.438, 0.348, 0.281, 0.229, 0.188, 0.158 and 0.125 inches. (The diameters of the mandrels were so chosen that if a leather specimen, 9 irons thick, was bent around successively smaller mandrels, the compression of the grain would increase in increments of 5 per cent. The calculation of the mandrel diameters is based on the assumption that the neutral axis of bending lies midway between the grain and flesh surfaces of the leather specimen.) All diameters shall have a tolerance of $\pm$ 0.002 inch. (This test is more easily performed if the mandrels are mounted in a bar as illustrated in Figure 1 of the method (P41) for "Grain Cracking".)

C. A bench vise or other means of securely gripping and supporting the mandrels.

D. A clamp of the mandrel jaw type having pressure surfaces at least as wide as the specimen. A small hand-screw wooden clamp meets the requirements.

4. Procedure

Depending on the type of leather, the thickness of the specimen shall be measured at its mid-point to the nearest 0.5 ounce or 0.5 iron.

Light Leather. The specimen, with its grain surface inward, shall be bent slowly around a mandrel held in the vise. First try a mandrel which it is anticipated will be too large to cause piping. If the specimen does not pipe, it shall be bent around successively smaller mandrels until piping occurs.

Heavy Leather. The specimen shall be bent into a "U", grain inward, placed in the jaws of the clamp, and over a mandrel too large to cause piping. The clamp shall be closed until the specimen contacts the mandrel lightly. As the clamp is closed, it shall be pushed forward to keep the bent portion of the specimen in contact with the mandrel. The specimen, still in the clamp, shall be lifted from the mandrel, examined and tested on successively smaller mandrels until piping occurs.

The diameter of the mandrel on which the specimen pipes shall be recorded.

5. Results

The range and average thickness of the specimens shall be reported to the nearest 0.5 ounce or 0.5 iron.

The maximum and minimum mandrel diameters on which individual specimens piped and the diameter of the mandrel on which the largest number of specimens piped shall be reported.

The compression of the grain at piping may be used as a measure of the resistance of the leather to grain piping as follows.

A. The percentage of grain compression at piping for the individual specimens can be calculated. It can also be obtained directly from the curves in Figire 2 of the method (P41) for "Grain Cracking" by reading as "grain compression" the percentages labeled "grain extension".

B. The average of the percentage grain compression for the individual specimens shall be the percentage grain compression of the sample and shall be reported.

The direction of the long axis of the specimen relative to the backbone shall be reported.

6. History and Status

A method for determining the resistance of leather to cracking and piping by bending the leather over mandrels was adopted as official in 1941.

GRAIN CRACKING (P41)

Cracking is defined as the rupture of the grain layer when a specimen of leather is bent through an angle of 180°. These cracks should not be confused with checking of the finish.

1. Scope

This method is intended for use on all types and thicknesses of leather.

2. Specimen

The specimen shall be a piece of leather, 1 inch wide and at least 4 inches long, cut with its long edges at least 1 inch away from any uncut edge of the unit. The direction of the long axis of the specimen relative to the backbone shall be recorded.

3. Apparatus

The Apparatus Shall Consist of

A. Thickness gages of the wedge or spring types as described in the method (P2) for measuring "Thickness of Units".

B. A set of mandrels consisting of 11 metal pins at least 4 inches long with diameters of 1.688, 1.063, 0.750, 0.563, 0.438, 0.348, 0.281, 0.229, 0.188, 0.158 and 0.125 inches. (The diameters of the mandrel were so chosen that if a leather specimen, 9 irons thick, was bent around successively smaller mandrels, the extension of the grain would increase in increments of 5 per cent. The calculation of the mandrel diameters is based on the assumption that the neutral axis of bending lies midway between the grain and flesh surfaces of the leather specimen.) All diameters shall have a tolerance of ± 0.002 inch. (This test is more easily performed if the mandrels are mounted in a bar as illustrated in Figure 1.).

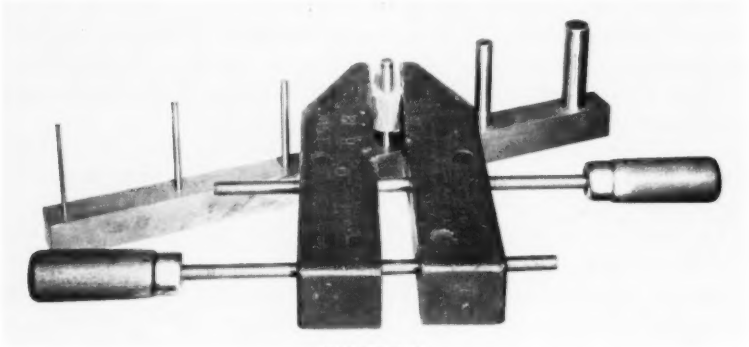


FIGURE 1.

C. A bench vise or other means of securely gripping and supporting the mandrels.

D. A clamp of the parallel jaw type having pressure surfaces at least as wide as the specimen. A small hand-screw wooden clamp meets the requirements.

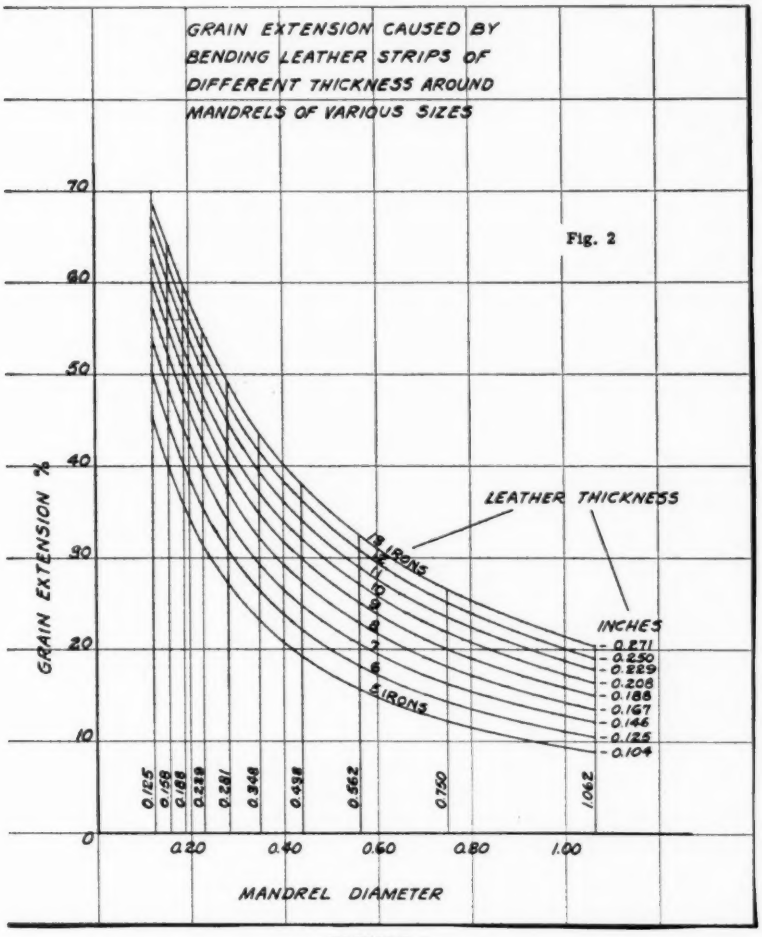


FIGURE 2.

1. Procedure

Depending on the type of leather, the thickness of the specimen shall be measured at its mid-point to the nearest 0.5 ounce or 0.5 iron.

Light leather. The specimen, with its grain surface outward, shall be bent slowly around a mandrel held in the vise. First try a mandrel which it is anticipated will be too large to cause cracking. If the grain does not crack, the specimen shall be bent around successively smaller mandrels until cracking occurs.

Heavy leather. The specimen shall be bent into a "U", grain outward, placed in the jaws of the clamp, and over a mandrel too large to cause grain cracking. The clamp shall be closed until the specimen contacts the mandrel lightly. As the clamp is closed, it shall be pushed forward to keep the bent portion of the specimen in contact with the mandrel. The specimen, still in the clamp, shall be lifted from the mandrel and tested on successively smaller mandrels until cracking occurs.

The diameter of the mandrel on which the grain of the specimen cracks shall be recorded.

5. Results

The range and average thickness of the specimens shall be reported to the nearest 0.5 ounce or 0.5 iron.

The maximum and minimum mandrel diameters on which the grain of the individual specimens cracked and the diameter of the mandrel on which the largest number of specimens cracked shall be reported.

The extension of the grain at cracking may be used as a measure of the resistance of the leather to grain cracking as follows.

A. The percentage extension of the grain at cracking for the individual specimens can be calculated or obtained directly from the curves in Figure 2.

B. The average of the percentage grain extension for the individual specimens shall be the percentage grain extension of the sample and shall be reported.

The direction of the long axis of the specimen relative to the backbone shall be reported.

6. History and Status

A method for determining the resistance of leather to cracking and piping by bending the leather over mandrels was adopted as official in 1941.

RESISTANCE TO CRACKING AT LOW TEMPERATURE (P42)

Resistance to Cracking at Low Temperature is defined as the ability of cold leather to withstand distortion and shock without cracking.

1. Scope

This method is intended for use on all light leathers. It is particularly applicable to coated leather such as upholstery leather.

2. Specimen

The specimen shall be a piece of leather 7 by 7 inches.

3. Apparatus

THE APPARATUS SHALL CONSIST OF

A. A specimen block made up of 1, a hardwood block 4 x 4 inches by 3/4 inch thick. At the geometric center of each of the 4 narrow sides of the block, a No. 6 round head woodscrew shall be driven to such a depth that the bottom of the head stands 1/8 inch away from the block, and 2, two rubber blocks each 4 inches square and 1 inch thick. These blocks shall be made of medium density sponge (foam) rubber and shall be placed on top of the wooden block. (An industrial sponge rubber which has a density such that a sheet one yard square and one inch thick shall weigh 22 pounds, may be used for this purpose.)

B. A refrigerated cabinet thermostatically controlled and capable of maintaining any temperature between 73° F. and – 65°F. with a tolerance of ± 2° F. The cabinet shall be large enough to hold the testing machine and allow for its operation while in the cabinet.

C. A testing machine is shown in Figure 1 or an equivalent device. It shall consist of the following parts: (1), a frame to support the weight-guiding tube, the weight-release shaft, and the specimen block, (2), a vertically-mounted weight-guiding metal tube at least 13½ inches long with an outside diameter of 3 inches and an inside diameter of 2 7/8 inches. The lower end of the tube shall be 3½ inches above the specimen block seat on the frame, (3), a 4-pound drop weight and chain of the design and materials shown in Figure 1, and (4), a weight-release shaft mounted on the frame so that one edge of the shaft is vertically over the center of the tube. The shaft shall be equipped with a pin, placed directly over the center of the tube, over which the weight chain can be hooked to suspend the weight, and a latch so arranged that the weight can be released from outside the refrigerated cabinet in which the test is performed.

4. Procedure

A 3/16 inch hole shall be punched in each corner of the specimen so that the outside-to-outside distance across holes on a diagonal of the specimen shall be 7 3/4 inches. The specimen shall be mounted with the grain side up on the foam rubber side of the specimen block with the holes in the specimen placed over the screws in the sides of the block.

The weight shall be lifted and the mounted specimen shall be placed in the machine. The height of the weight shall be adjusted so that its lowest point is 9½ inches above the specimen. (This height of drop is used so that that when allowance is made for unknown machine losses, the weight will hit the specimen with a velocity of approximately 7 ft. / sec. and an energy of approximately 3 foot pounds.) The testing machine, ready for operation, shall be placed in the cold cabinet. When the desired cabinet temperature

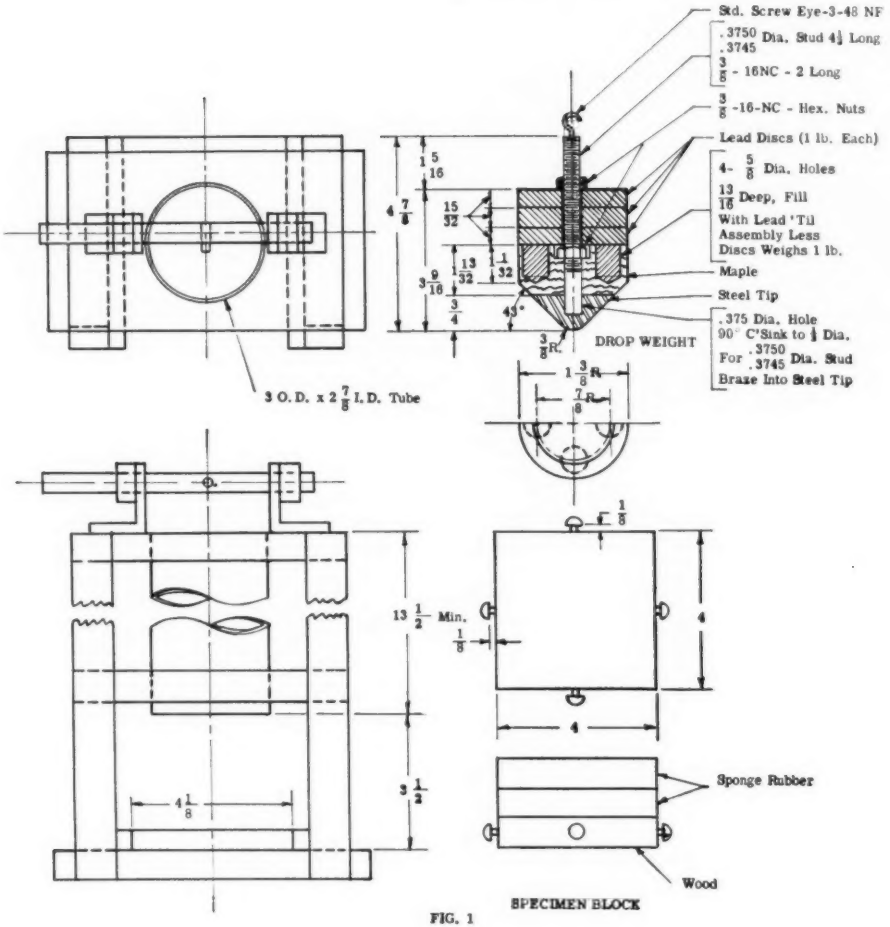


FIG. 1

(usually 20° F.) has been reached and maintained for at least 30 minutes, the machine shall be tripped from outside the cabinet, permitting the weight to fall on the specimen. The specimen shall be removed and examined for cracks. If the specimen has not cracked, it shall be returned to the testing machine and the drop weight again lifted to position. The temperature of the cabinet shall be reduced 25° F. (usually to -5° F.). When the new cabinet temperature has been reached and maintained for 30 minutes, the machine shall be tripped and the specimen shall be re-examined. The specimen shall

be tested and examined at each successive 25° F. reduction in temperature until cracking occurs.

5. Results

The temperature at which the specimen cracked shall be reported.

If the weight is dropped from a height other than 9½ inches, the height of the drop shall be reported.

6. History and Status

An "Impact Method for Measuring the Resistance of Coated Light Leather to Cold Cracking" was adopted as official in 1944. Experience with this method showed it did not work satisfactorily on a wide variety of leathers and it was removed as a method in 1948.

STIFFNESS (P43)

Stiffness, or resistance to bending, is defined as the force or moment required to bend a specimen through a given angle.

1. Scope

This method is intended for use on all types of leather.

2. Specimen

The specimen shall be 0.50 inch wide and at least 1.5 inches longer than the testing span. Its length shall not exceed 4 inches. Whether the unit from which the specimen was taken is the full thickness of the skin or has been split shall be recorded.

3. Apparatus

The Apparatus Shall Consist of

A. A Tinius-Olsen 5-inch-pound stiffness tester, as shown in Figure 1. (As far as is known, this is the only commercial device which meets the requirements of this test. If other satisfactory devices are developed, their use will be allowed.) The device has the following elements.

A horizontally mounted motor-driven shaft to one end of which a vise for clamping the specimen is fixed. The shaft carries an adjustable pointer which passes over a scale on the pendulum indicating the relative angular motion of the vise and the pendulum, and thus, the angle through which the specimen is bent.

A balanced circular pendulum mounted on knife edges which are in line with the center line of the shaft. The pendulum carries

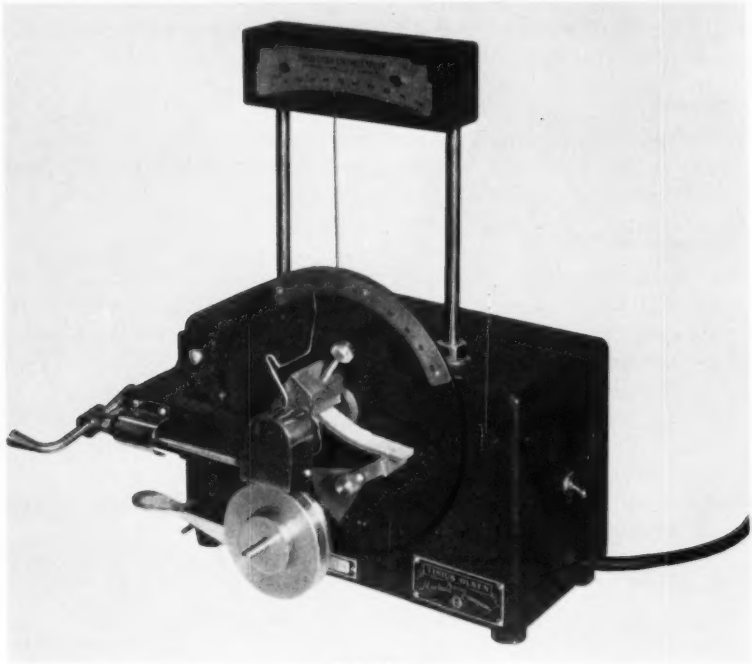


FIGURE 1.

1. A pin on which weights can be hung to unbalance the pendulum.

2. A fulcrum against which the unclamped end of the specimen presses during the test. The position of the fulcrum can be changed to get spans of 0.25, 0.5, 1.0, and 2.0 inches.

3. A circulate scale graduated from 0 to 90 degrees used as described above.

4. A pointer which passes over a load scale on the frame to indicate the displacement and hence the torque of the pendulum.

A load scale on the frame which is graduated in per cent of the moment of the pendulum when it has maximum displacement, 100 per cent, representing a pendulum displacement of 30 degrees.

A set of weights which will give maximum pendulum torques at 30 degrees displacement of 0.1 to 5.0 inch-pounds in increments of 0.25 inch-pound.

Equipment so the motor can be thrown in and out of gear, the vise can be rotated by hand for resetting to the original position, and the pointers can be set at zero on the scales.

B. A dead-weight type thickness gage as described in the method (P3) for "Thickness of Specimens".

C. A steel scale graduated to 0.2 inch.

4. Procedure

The width (w) of the specimen shall be measured on the grain side and on the flesh side to the nearest 0.02 inch at three places equally spaced along that part of the specimen which will constitute the free beam in bending. The average of the readings shall be used as the width of the specimen.

The thickness (t) shall be measured to the nearest 0.001 inch in three places on the long axis equally spaced along that part of the specimen which will constitute the free beam in bending. The average of the readings shall be used as the thickness of the specimen.

The machine shall be set with the proper span (L) and pendulum weights (P). (It is suggested that the span and the pendulum weights be selected so that the reading on the load scale will be between 25% and 75%, when the specimen is bent through an angle of 50 degrees.) The specimen shall be mounted in the clamp so that the grain will be convex when the specimen is bent. The shaft shall be turned by hand until the free end of the specimen just touches the specimen loading fulcrum on the pendulum. The pointers shall both be adjusted to read zero. The motor shall be engaged and the shaft shall continue to turn until the specimen has been bent through an angle of 50 degrees (angle a). The per cent bending moment (m) shall be read from the load scale and recorded.

If, for any reason, a specimen does not bend in a smooth curve, it shall be discarded and replaced by another specimen.

5. Results

The results may be reported in any or all of the following ways.

A. For comparative purposes the total of the weights on the pendulum and the percentage bending moment may be reported.

B. The true bending moment in inch-pounds required to bend the specimen through a given angle shall be obtained by multiplying the per cent bending moment (m) by the total of the pendulum weight (P).

$$\text{The true bending moment, } M = mP.$$

C. The force on the unclamped end of the specimen required to bend the specimen shall be calculated by dividing the true bending moment (M) by the length of the span (L).

$$\text{The force, } F = \frac{M}{L}$$

D. A relative stiffness factor can be obtained by using the following equation.

$$\text{Relative stiffness factor, } E = \frac{ML}{wt^3a}$$

where

M is the true bending moment in inch-pounds.
L is the length of the span in inches.
w is the width of the specimen in inches.
t is the thickness of the specimen in inches.
a is the angle in degrees through which the specimen was bent.

The angle through which the specimen is bent shall be reported.

The values of any of the above factors obtained from the individual specimens shall be averaged to obtain that value for the sample.

The average of all the thickness readings taken on the specimens shall be the thickness of the sample and shall be reported.

6. History and Status

This method was adopted by the Association as an official method in 1942.

STAINING (P44)

Staining is defined as the transfer, at an elevated temperature, of coloring matter or other substances from leather to a wet materials, such as water saturated cloth, which is pressed against it.

1. Scope

This method is intended for use on all types of leather.

2. Specimen

The specimen shall consist of two pieces of leather 4 inches by 4 inches.

3. Apparatus and Materials

The Apparatus and Materials Shall Consist of

A. Cloth pads approximately 2 inches square consisting of 4 layers of starch-free, white, bleached cotton print cloth with an 80 by 80 thread count. If a comparison standard is used, two pads are required.

B. A standard comparison leather with acceptable staining characteristics.

C. Four pieces of plate glass 6 inches by 6 inches by 1/4 inch.

D. A thermostatically controlled air oven capable of maintaining a temperature of 70° C. within ± 2° C.

4. Procedure

A piece of the test leather with its grain side up shall be placed on a glass plate. The cloth pad shall be thoroughly wet by immersion in distilled water

and placed on the piece of leather. The second piece of leather shall be placed with its grain side down on the pad. The second glass plate shall be placed on top of the leather. Care shall be taken to place each piece centrally with the preceding piece and the edges of all pieces shall be parallel.

The assembled test unit shall be placed in an oven at 70° ± 2° C. for 1 hour, after which it shall be removed and allowed to cool.

The test unit shall be opened and the pad shall be examined for staining.

If a comparison standard is used, a unit using the comparison pieces shall be prepared and tested as outlined above.

5. Results

If no comparison standard is used, the amount of staining shall be judged and reported as

A. Little staining

B. Appreciable staining

C. Heavy staining

If a comparison standard is used, the pad used with the test leathers shall be compared with the pad used with the comparison leathers.

6. History and Status

A method for measuring the "Resistance of Colored Leather to Bleeding" was adopted as official in 1944 and has been retitled.

COMPRESSIBILITY OF LEATHER (P45)

Compressibility is defined as the percentage change in thickness, produced by subjecting a leather specimen to a given compressive force for a given length of time.

1. Scope

This method is intended for use on all kinds of leathers. It is particularly applicable to heavy leathers such as sole leather.

2. Specimen

The specimen shall be a piece of leather 2 ± $^1/_{16}$ inches by 2 ± $^1/_{16}$ inches. Care shall be taken to cut the specimen so that the edges will be normal to the surface.

3. Apparatus

The Apparatus Shall Consist of

A. A compression testing machine having a capacity of at least 15,000 pounds, either hand or power driven. The applied load shall be indicated on

a dial, scale, or chart. The machine shall be equipped with one spherical head or other means to insure a uniform pressure distribution over the specimen.

B. A dead-weight type thickness gage as described in the method (P3) for "Thickness of Specimens".

C. A steel scale graduated to 0.02 inch.

D. A timing device, either a watch or other timer which can be read to 5 seconds.

4. Procedure

The thickness shall be measured to 0.001 inch at the middle of each side approximately 0.5 inch from the edge. The four values shall be averaged to give the original thickness (t_1) of the leather.

The length of the four edges of the specimen shall be measured on the grain side to the nearest 0.02 inch. The length of opposite edges shall be averaged to obtain two values which shall be multiplied together to give the area (A) of the specimen.

The specimen shall be placed in the machine and a load of 3000 ± 100 pounds per square inch, (3000 ± 100)A, shall be applied. The speed of the machine head shall be such that the load is applied at a rate of approximately 8000 pounds per second. After the maximum load is reached, it shall be maintained by machine adjustments for 3 minutes.

The load shall be removed and thickness measurements shall be made immediately at the same four points at which the original thickness measurements were made. These measurements shall be averaged to give the compressed thickness (t_2) of the leather.

5. Results

The compressibility of the specimen shall be determined by either formula A or B as follows.

$$\text{A - Compressibility, on basis of compressed thickness} = \frac{t_1 - t_2}{t_2} \times 100$$

$$\text{B - Compressibility, on basis of original thickness} = \frac{t_1 - t_2}{t_1} \times 100$$

Compressibility shall be computed to the nearest 0.1%.

The compressibility of the sample shall be obtained by averaging the compressibilities of the individual specimens.

6. History and Status

A method for measuring the compressibility of leather, which had been conditioned at 70° F. and 65% relative humidity, then wet with 10 ± 2% of water and stored at 75° ± 5° F. and 90–100% relative humidity for 16 hours,

was adopted as provisional in 1944. The method was changed in 1947 to require conditioning of the leather at 73° ± 2° F. and 50 ± 4% relative humidity with no added water and was made official in 1949.

CROCKING (P46)

Crocking is defined as the transfer of coloring matter or other substances from leather to wet (damp) or dry cloth rubbed against it.

1. Scope

This method is intended for use on any type of leather.

2. Specimen

The specimen shall be a rectangular piece of leather at least 2 inches by 5 inches. Separate specimens are required for dry and wet crocking. To avoid cutting specimens, the test can be run at two separate places on the unit by placing the edge of the unit on the crockmeter.

3. Apparatus

The Apparatus and Material Shall Consist of

A. A crockmeter as shown in Figure 1 or equivalent.

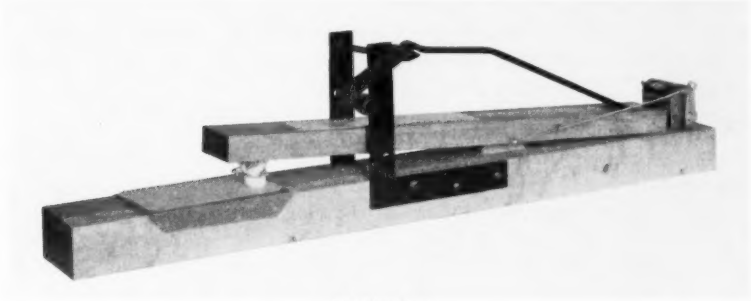


FIGURE 1

(The Crockmeter shown in Figure 1, and the AATCC Color Transference Chart, may be obtained from the Secretary of the American Association of Textile Colorists and Chemists (AATCC), Lowell Institute, Lowell, Mass. The black cloth may be obtained from the Secretary of the ALCA, and the white cloth, cut into 2 x 2 inch squares, and packaged in lots of 1000, from Testfabrics, Inc., 55 Van Dam Street, New York, N. Y.) The device consists of a flat base covered with abrasive cloth on which the specimen rests during the test. Attached to the base is a slide piece which can be operated by a

crank to slide a distance of 4½ inches back and forth over the base. The slide piece is equipped with a cylindrical brass finger, ⅝ inch in diameter. The end of the finger is flat, except that the edges are slightly rounded. A 2-inch square piece of cloth is placed over the finger, avoiding wrinkles on the flat end surface, and held in place with a spiral clip. The slide is loaded so that the finger presses on the specimen with a load of 3 ± ½ ounces.

B. Starch-free, unfinished, combed lawn with a 96 by 100 thread count and a weight of 5.3 yards per pound. For testing colored leathers, white bleached cloth (lawn) shall be used. For testing white leathers, colorfast black cloth shall be used.

C. A comparison standard which shall be a piece of leather with the desired crocking characteristics. For development purposes or when otherwise agreed upon, the AATCC chart for Measuring Transference of Color may be used as a comparison standard.

4. Procedure

Dry Test. The specimen shall be mounted flat on the base of the testing machine with its long dimensions in the direction of the motion of the finger. The cloth used for testing colored leathers shall be white, that used for white or light-colored leathers shall be black. The piece of cloth, 2 inches square, shall be conditioned for at least 2 hours in the standard atmosphere, then placed on the end of the finger with the weave of the cloth oblique to the direction of rubbing. The finger with the cloth cover shall be placed on the specimen and the machine shall be operated for 10 complete turns of the crank at a rate of 1 turn per second. When a comparison is required, a standard specimen shall be tested in the same manner.

Wet Test. A new specimen and white or black cloth shall be placed on the testing machine as described above under "Dry Test". Before placing the cloth on the finger, it shall be wet with distilled water, then wrung or extracted until its moisture content is between 75 and 100 per cent of the weight of the cloth before wetting. The rubbing test shall be carried out as described under "Dry Test". At the end of the test, the cloth shall be removed and dried in air. When a comparison is required, a standard specimen shall be tested in the same manner.

5. Results

If no comparison standard is specified, the colorfastness of the specimen shall be classified as follows.

Good—No appreciable staining of either the wet or dry cloth.

Fair—Appreciable staining of wet cloth but no appreciable staining of dry cloth.

Poor—Appreciable staining of dry cloth.

If a comparison standard is used, the cloth used on the test specimen shall be compared with the cloth used on the standard specimen.

If the AATCC chart is used for comparison, the cloths used on the test specimens shall be compared with the chart.

The results of the dry and wet tests shall be reported separately.

6. History and Status

A method for measuring the "Resistance of Leather to Crocking" was adopted as provisional in 1946.

The Chemistry of Vegetable Tannins

VII. Acetyl Values

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In an article published in this *Journal* **48**, 133 (1953) by D. G. Roux, exception is taken to the method of determining acetyl values by alkaline hydrolysis as applied to the substances discussed in the current series of papers by the present authors and coworkers.

The thesis that the difference in acetyl contents is due to the difference in analytical methods has much merit. The authors have not been unaware of this possibility and its implications.

In the following discussion three methods of hydrolysis will be considered:

1. Alkaline hydrolysis.

The method of alkaline hydrolysis consists in refluxing the substance with 4 per cent sodium hydroxide in 50 per cent methanol for 30–90 minutes, removal of the alcohol, acidification by sulfuric acid and magnesium sulfate, distillation of the acetic acid and titration of the acid [18,19].

2. Acid hydrolysis.

This is essentially the reverse of the previous method. Hydrolysis is carried out with sulfuric acid and magnesium sulfate. The acetic acid liberated is distilled and titrated.

3. Trans hydrolysis.

This is the method of Matchett and Levine [1] and involves transesterification in the presence of acid and alcohol, distillation of the ethyl acetate formed, hydrolysis by alkali and titration.

The principal arguments advanced in favor of trans hydrolysis follow:

1. Alkaline hydrolysis of the original tannins gave acetyl values [2,12].

2. Fisetin gave an acetyl value [2] by alkaline hydrolysis.

3. Acetylation of wattle did not give high acetyl values by trans hydrolysis [3].

4. Autoclaving d-catechin, pyrogallol and wattle with alkali gave acids [4].

5. Acetylhaematoxylin yielded formic acid with alkali [5].

6. Trans hydrolysis standardized against penta-acetyl d-catechin and triacetylpyrogallol gave quantitative results.

Let us now consider the objections to accepting the results of acid or of trans hydrolysis rather than those obtained by alkaline hydrolysis.

1. Alkaline hydrolysis of tannins gave acetyl values whereas infra-red spectrography showed a negligible or absent keto group. In certain cases, however, both acid and trans hydrolysis gave acetyl values also, viz.:

Substance	Trans	% Ac Acid	Alkali
Quebracho A	0.91	0.00 (insol.)	8.83
Mangrove A		0.00	5.53
Mangrove B	3.32	1.59 (insol.)	8.29
Wattle	1.11		3.87

2. Failure of the substance to completely dissolve in acid reagents casts doubt on the results.

3. Decomposition by alkali of a tannin to give acid does not necessarily apply to its acetyl derivative since we are not dealing with the same molecule.

4. Reasoning from penta-acetyl d-catechin to tannins is precarious. Freudenberg and others found it not possible to remove the fifth acetyl group from penta-acetyl d-catechin with sodium sulfite, sodium carbonate, piperidine, aniline or tannase [6]. Either the penta-acetyl d-catechin was recovered unchanged or the substance was destroyed, In such a case alkali gave too low a result. The present authors were unable to obtain it in pure enough form to use it as a standard.

5. Autoclaving of d-catechin, pyrogallol and wattle with alkali at 240°F is a severe treatment. It is comparable to using strong alkali at lower temperatures and naturally breaks down the molecules. It cannot be compared to the alkaline hydrolysis which employs 4 per cent alkali in 50 per cent methanol at 65°C under reflux.

6. It is true that phenols are unstable in the presence of alkali, but the oxidation products are not necessarily acidic. Phenolic acids, however, show some stability since β-resorcylic, and protocatechuic acids can be recovered after alkaline fusion of tannins. The instability of phenolic nuclei evidently depends on the particular molecule.

7. Does it not seem more than fortuitous that duplicate alkaline hydrolyses gave acetyl values which were in agreement? Would this occur if the high values came from decomposition?

8. Is it not remarkable that the values obtained are analytically correct for the peracetylated derivatives? In no case did the number of acetyl groups exceed the number of oxygen atoms in the original tannin. One might have expected a constantly increasing value with time.

Mangrove (B) acetylated:

% Ac	Alkaline saponification time (min.)
30.12	30
48.25	90
47.83	90

9. Three acetylations were required to raise mangrove and wattle to top values. Mangrove gave an octa-acetyl compound with only two acetylations. Why did not this substance show excess acetyl? If they are difficult to acetylate, may they not be difficult to deacetylate?

10. Freudenberg considered the opening of the ether ring in catechin a distinct possibility. He postulated that the higher acetyl content of acetylated catechin phlobaphenes was due to this cause [7, 11].

11. Formic acid cannot be the cause of high acetyl values since the subsequent acidification with sulfuric acid and magnesium sulfate, followed by distillation, destroys any formic acid formed.

12. Acetylation in pyridine at room temperature does not affect the 5-hydroxyl groups in polyhydroxy flavones [8]. A temperature of 100° is required. The use of acetic anhydride and sulfuric acid gives complete acetylation with flavanones [9].

Possibly some of the hydroxyl groups in tannins are difficult to acetylate and once acetylated difficult to deacetylate. Is it possible that the peracetylation has suceeded, but that the trans hydrolyses have been incomplete?

13. Deacetylation of 7 acetoxy, 2,3,5, trimethyl chromone and similar compounds required the use of concentrated sulfuric acid [10]. Would trans hydrolysis have succeeded in removing all the acetyl groups?

14. Acetic anhydride and pyridine have been found unsatisfactory for acetylation of tertiary alcoholic groups. Only a 10% reaction was reported when a temperature of 100°C. for one hour was employed [14]. The use of acetyl chloride instead of the anhydride may increase the yield to 30–40 per cent of the theoretical [15]. The difficulty in acetylation may indicate a similar difficulty with deacetylation.

15. Tertiary alcohols have been completely acetylated by using acetic anhydride and sodium acetate [16]. Since quaternary ammonium salts are

more effective than sodium acetate[17] the present authors have employed this procedure followed by the acetyl chloride-organic base treatment.

16. According to Clark, analysis for acetyl groups by trans hydrolysis of small quantities fails because the ethyl acetate cannot be completely hydrolyzed without employing a standard alkali solution too strong to yield accurate reulsts[18], [19].

17. The methyl ester of triacetyl cholic acid must be hydrolyzed with methanolic sodium hydroxide to remove the acetyl groups[20].

In view of the foregoing considerations, further experimental work has been carried out to clarify the subject. The trans hydrolysis method was tried out on some known recrystallized compounds dried to constant weight. The average results follow:

Substance	Calc. Ac	Found Ac		
		Trans	Acid	Alkali
Pyrogallol triacetate	51.19	47.66	25.57	51.25
Hydroxyhydroquinone triacetate	51.19	49.97	43.91	50.18
Pentacetyl quercetin	42.00	39.41	0.00	37.96
d-Catechin	0.00	0.95	0.00	0.00

The above data indicate that trans hydrolysis is not quantitative in all cases. The d-catechin was prepared from gambier and specially purified, showing a rotation:

$$[\alpha]_D^{27^\circ C} = +18^\circ$$

$$C = 3.33\% \text{ in } 50\% \text{ acetone.}$$

It supersedes the result previously reported on a commercial sample[2].

In order to observe the results with an acetylated tannin, acetylated quebracho was analyzed. This white product showed no hydroxyl absorption in its infra-red spectrogram. It naturally had a very powerful keto absorption. Analysis by trans hydrolysis indicated an average content of 31.55 per cent acetyl. This compared with 46.66 per cent by alkaline hydrolysis. The residue from the trans hydrolysis was tested for residual acetyl content by preparing its infra-red spectrogram. It clearly showed a significant keto absorption. This indicated that all of the acetyl groups had not been removed by trans hydrolysis. The only other explanation was a possible formation of some other keto groups during the hydrolysis. This is unlikely since quebracho itself after trans hydrolysis treatment and recovery showed no increase in

the very small keto absorption originally present in its infra-red spectrogram.

A further indication that 31.55 per cent is far too low a figure for the acetyl content is the fact that no hydroxyl absorption was shown by the acetylated quebracho. This acetyl figure only corresponded to six acetyl groups, viz.:

Calc.	$C_{31}H_{22}O_5$ $(O\,Ac)_6.H_2O$				
	C 60.99	H 5.00	Ac 30.50		
Found	C 61.48	H 4.54	Ac 31.71		
	61.77	4.68	31.37		trans
			31.57		hydrolysis

This would require the remaining five oxygen atoms to be present as ether linkages, for if any had been present as hydroxyl groups they would have been observed. They could not be present as keto groups since the original quebracho showed negligible keto absorption. It thus appears necessary to conclude that trans hydrolysis of acetylated quebracho does not give the true acetyl value.

Let us now consider the acetyl content of deacetylated quebracho. This product was prepared by deacetylating quebracho with potassium acetate [13] and analyzed as follows:

Calc.	$C_{31}H_{25}O_7$ $(O\,Ac)_3$–$1.5H_2O$			
	C 60.90	H 5.11	Ac 17.70	
Found	C 60.94	H 4.85	Ac 18.70	(alkaline
	61.09	4.81	18.49	hydrolysis)

By trans hydrolysis an acetyl content of 8.60 per cent was obtained. Inspectiont of the infra-red spectrogram of the residue revealed that some keto absorption still remained. This showed that trans hydrolysis failed to remove the acetyl groups completely.

The same procedure was followed with wattle. Acetylated wattle gave a value of 33.08 per cent acetyl by trans hydrolysis whereas the value by alkaline treatment was 48.01 per cent. Deacetylated wattle gave a value of 0.62 per cent (ca.) by the former method and a value of 18.42 per cent by the latter. In both cases the residues obtained from the trans hydrolyses showed the presence of keto absorption and thus incomplete removal of the acetyl groups.

Analyses of acetylated mangrove (B4) [2] gave a value of 31.85 per cent by trans but a value of 35.56 per cent by alkaline hydrolysis. On reacetylation this product (B5) [2] gave values of 31.37 per cent and 48.04 per cent respectively. It is difficult to understand why the increased acetyl content is detected only by the lattermethod. It appears that trans hydrolysis may be limited to acetyl groups substituted on only certain types of hydroxyl groups.

From the foregoing we are forced to conclude that trans hydrolysis as preformed by us gives values which are too low.

The question now remains—what of the acetyl values found for the original tannins? We see no way in which the acetyl values for acetylated tannins can be reliably corrected for apparent acetyl contents of the unacetylated tannins[2].

Let us still assume that the acetyl value for acetylated quebracho as found by alkaline hydrolysis is too high. What is the effect on the carbon-hydrogen values if we assume, for example, that it has only nine rather than eleven acetyl groups present? Actually the effect is very small, and the analyses are still satisfactory, so that no effect is produced on the basic formula, viz.:

Calc.	$C_{21}H_{19}O_2$ (O Ac)$_9$			
	C 61.64	H 4.86	M.W.	954.8
Found	C 61.48	H 4.54	M.W.	1,020
	61.77	4.68		

The molecular weight determination causes us to prefer the original undecacetyl compound whose calculated molecular weight is 1,039.

The same is true of wattle. If we assume only ten acetyl groups are present, the carbon-hydrogen values are still satisfactory:

Calc.	$C_{30}H_{16}O_2$ (O Ac)$_{10}$	
	C 60.11	H 4.64
Found	C 59.90	H 4.48
	60.18	4.18

The acetyl values of the acetylated tannins, however, obtained by alkaline hydrolyses, point to and give the correct values for peracetylated derivatives.

The data referred to above are collected in the following table.

	% Ac		
	Trans	Acid	Alkali
Acetylated quebracho	31.55	0.84 (insol.)	46.66
Acetylated wattle	33.08		48.01
Acetylated mangrove (B4)	31.85		35.56
Acetylated mangrove (B5) (B4 reacetylated)	31.37		48.04
Deacetylated quebracho	8.60	5.90 (sol.)	18.60
Deacetylated wattle	0.62 (ca.)	0.99 (insol.)	18.42
Deacetylated mangrove (A)		0.00 (insol.)	15.68

Summary

Acetyl values of three purified tannins and their derivatives have been obtained by different methods and the data compared. Analysis of the results

in the light of infra-spectrograms of the residues from the acetyl determinations in conjunction with a consideration of past literature on the subject indicates that alkaline hydrolysis gives the most dependable values.

Acknowledgment

The authors wish to acknowledge the courtesy of Dr. Roux in allowing them to see the manuscript of his paper before publication.

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Received, December 14, 1952.

A Recording Multiple-Sample Shrinkage Temperature Instrument

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Introduction

The measurement of the shrinkage temperature of various types of leather has gained increasing interest and importance for those engaged in practical tannery quality control[1] and for research workers who are interested in the mechanisms of tanning and the development of new or modified tannages.

In general, for any one type of tannage, the shrinkage temperature measurement provides information about the degree and uniformity of the tannage. To the research worker, the shrinkage temperature will indicate the relative ability of a tannage to withstand hydrolytic attack with its consequent washing out of tanning material and loss of leatherlike properties.

The method generally in use at present [2] is time consuming, and requires the fairly constant attention of a technician throughout the determination. As a result, this measurement, on 10 to 12 samples per day, would almost completely engage the time of one technician. Even if a tannery should be willing to assign a technician to this task alone, the number of samples involved may be too few to meet the needs of a modern repetitive program of statistical quality control. Experience at this Laboratory has indicated that even a modest scale research project investigating varied conditions of combined tannages may very rapidly accumulate a large number of samples whose shrinkage temperatures would be worthwhile to know. However, normal limitations of personnel and sensible economy prevent all the desired measurements from being made. The shrinkage temperature is also an essential determination in the worthwhile technique of layerwise analysis for research and quality control. However, the layerwise analyses multiply the number of samples, and they therefore encounter the same limitations already noted. An instrument has been designed and constructed which overcomes the objections discussed above, while yielding results of equal accuracy to those obtained by the official A.L.C.A. instrument and procedure [2].

The new instrument is capable of determing the individual shrinkage temperature values of six samples simultaneously. Furthermore, once the samples are clamped in the instrument and the operation is started, the constant attendance of the technician is not required. The individual shrinkage temperature values may be read at any time after shrinkage on maximum reading thermometers (similar to clinical fever thermometers), which are mechanically removed from the water bath and suspended in the air at the moment that shrinkage commences.

Discussion

An over-all photograph of the instrument is shown in Figure 1. This particular instrument can handle six samples at once; the same design principles permit the instrument to be constructed so that it can handle any lesser or greater number as desired while retaining the individual automatic recording features.

The samples are held in place by a system of clamps, supports, pulleys, and counterweights, which are essentially similar to those used in the conventional shrinkage meter [2]. On the horizontal supporting rod that holds the sample pulleys, there is also a plastic rod and counterweighted maximum reading thermometer for each sample. The housing above the pulleys con-

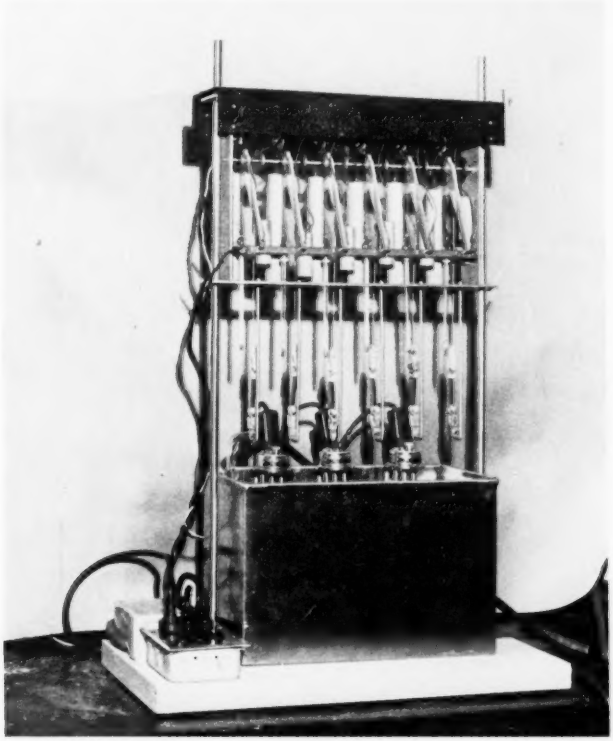

FIGURE 1.

tains a relay-solenoid assembly which will be discussed subsequently. This housing, the clamps, thermometers, etc., all constitute an assembly which may be raised or lowered on the two vertical rods. A rectangular water bath is shown below which is fitted with immersion heaters and a circulating pump. The upper assembly may be lowered until its lower supporting bar rests on the rim of the bath, at which time the thermometer bulbs and the samples will be sufficiently immersed in the bath.

Figure 2 is a close-up photograph of the essential mechanism of the instrument. The operating sequence of the instrument may be followed if one considers the second sample from the left (No. 2) to represent a shrunken condition.

The upper sample clamp is fixed to a thin metal rod which passes freely through a hole in the lower supporting bar. A pice of nylon cord is tied to this rod and passed over a pulley and tied to a counterweight. This pulley

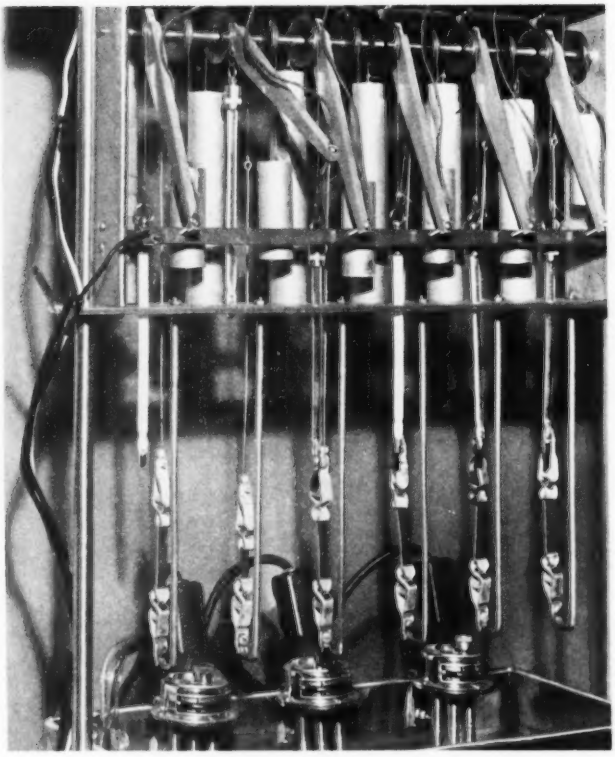

FIGURE 2.

is mounted on a bushing which rides freely on the main horizontal supporting bar. The lightweight plastic rod is mounted on the same bushing. The other end of this plastic rod contains a screw whose head is connected to a wire leading to one side of a six volt electrical source and relay assembly. The tip of the screw makes contact with the long horizontal bus bar which is electrically connected to the other side of this six volt circuit.

The pulleys which support the thermometers have a small notch in their circumference. The apex of a "V" shaped bar fits into this notch from above and prevents the rotation of the pulley. This bar is the plunger of a solenoid. When the solenoid is energized, the bar is elevated, and the thermometer pulley is released. The greater weight of the counterweight then pulls the thermometer out of the bath.

The following is a brief description of the operating sequence. The bath is filled; the samples are clamped into position; the upper assembly is then

lowered into position. The thermometers should be immersed and their pulleys locked by the "V" bar. The plastic rods are set so that the screw tips are all in contact with the bus-bar. The heaters and pump are turned on and the bath temperature rises uniformly.

The sample usually elongates or swells slightly at temperatures just below shrinkage temperature. This tends to cause a clockwise rotation of the sample pulley and the plastic rod. However, the other end of the plastic rod rests against the bus bar and the pulley cannot turn. It remains in position and the nylon cord merely slips over the pulley due to the pull of the counter-weight. When the sample starts to shrink the sample pulley is free to move in a counter-clockwise direction, raising the plastic rod and breaking its electrical contact with the bus-bar (see sample No. 2). Since the length of the plastic rod is several times the diameter of the sample pulley, a relatively small travel of the circumference of the sample pulley results in a much greater travel of the end of the plastic rod and thereby assures a prompt and positive breaking of the contact, unless the samples are shrinking in an extremely sluggish manner.

The breaking of the contact activates a relay-solenoid combination which raises the "V" bar out of the notch in the thermometer pulley, and causes the thermometer to rise out of the bath. This sequence of events occurs for each individual sample so long as it shrinks at or below the boiling point of the bath medium. The individual thermometer readings may then be noted when convenient.

A few refinements add to the convenience of the instrument. Standard 1-kilowatt heaters are used which also contain built in thermostats which will shut off the power to the heaters at any desired temperature such as the boiling point of the bath liquid, so that it will not boil away vigorously if left unattended for a prolonged period. The circulating pump is fitted with "T" connections in such a manner that it may be used to empty the bath and to refill it from a storage jar or the water tap.

An example of the accuracy and reliability of the instrument is illustrated in Table I below. Twelve pairs of samples were taken from scattered areas of a bend. The shrinkage temperature value of one sample of each pair was determined individually by the official instrument and procedure. The shrinkage temperature value of the other sample of each pair was determined in the new automatic instrument, in two groups of six samples each. The results are shown in Table I, and they indicate that there is no appreciable difference between results obtained with the conventional instruments and the new instrument.

The instrument is necessarily sensitive for its intended purpose, and therefore must be used with some intelligence and care; if it is so used, the desired advantage in economy of time and personnel and the acquisition of greatly augmented information may be realized. The cost of a six-sample

TABLE I

Shrinkage Temperature

Position	Official	Automatic
1	92.0	92.5
2	91.5	91.0
3	92.5	93.0
4	91.5	91.0
5	92.0	92.0
6	92.0	91.0
7	89.5	90.0
8	90.5	90.0
9	91.5	91.5
10	91.0	90.5
11	91.0	91.0
12	92.0	91.5

unit of this type is reasonable in relation to the cost of the conventional instrument as well as permiting a reduction in personnel time. It is commercially available

Additional information will be supplied to all who may be interested.

Acknowledgment

Appreciation and acknowledgment are gratefully extended to Mr. F. L. Collins of the International Shoe Company for supplying some of the initial suggestions for this work. Similar acknowledgment is made to Mr. Wesley Love and Mr. Dave Edwards, graduate students in the Physics Department of this University for their part in the design and construction of the instrument. The interest and encouragement of Dr. Fred O'Flaherty, and the material support of the Tanners' Council of America and its Sole Leather Group is most heartily acknowledged.

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Received October 15, 1952.

ABSTRACTS

Influence of Application of Fats on the Properties of Shoe Upper Leathers. F. Stather and H. Herfeld. *Ges. Abhandl. deut. Lederinsts. Freiberg/Sa.* No. 3, 57 (1950). The literature is reviewed (29 references). The present paper deals with the effects of the amount and kind of fat on physical properties (tensile strength, stretch, stitch tear, slit tear, water absorption, rate of wetting, rates of transmission of water, air and water vapor, and thermal conductivity) of stuffed, vegetable-tanned upper leather ("Fahlleder") and fat-

liquored chrome leather. Each test was made on the butt area of a whole skin, processed by commercial methods before and after the application of oil. Each comparative test was made on material from one skin. When only two treatments were involved, the butt was cut into quarters, and diagonally opposite quarters were treated alike. For three treatments, the central 1/3 was given treatment 1, and each pair of diagonally opposite 1/6th's was given a different treatment. The areas sampled were as nearly adjacent as possible. The vegetable leather was stuffed by hand application of the stuffing mixture to the flesh side, followed by hot drumming. The standard stuffing mixture consisted of equal parts (on fat basis) of train oil, tallow and degras. The standard amount given was 20% on the wrung weight. The normal fat content of the finished leather was 18-22% (H_2O = 14%). The chrome leather was fat-liquored. The standard fat liquor was sulfated castor oil, either lightly or heavily sulfated (degrees of sulfation, i.e., % of total ricinoleic acid molecules sulfated, 17 and 46% respectively). The standard amount given was 2.5% actual oil on the shaved weight, and % total fat (extractable with petroleum ether + bound) in leather (14% water) varied from 4.7 to 6.1 in different tests. Variation in treatments, and resulting effects on the measured physical properties, were as follows: *Vegetable leather.* (1) A 30% reduction in quantity of fat given lowered the tensile, stitch tear, and slit tear strengths, and the % stretch at break by from 4-5 to 9%; water uptake (24 hrs.) increased by 22%, wetting time (grain) decreased 18%, air transmission coefficient increased 22%, water vapor transmission coefficient increased 8%, and thermal conductivity decreased 8%. A 30% increase in given fat affected all these properties in the opposite directions, but the differences from the standard were only about a fourth as great. (2) Replacement of 45% of the train oil by tallow had a negligible effect on strength, decreased the water absorption by only 7%, increased the wetting time by 16%, and lowered the air- and water vapor transmission coefficients by 19 and 16% respectively. Replacement of 45% of the tallow by train oil increased the strength by 6-11%, increased the water uptake by 12%, decreased the wetting time by 12%, and increased the air and water vapor transmission coefficients by 12 and 3% respectively. (3) Replacement of 41% of the degras by equal parts of tallow and train oil lowered the water absorption by 7%; other properties were not appreciably affected. Replacing 24% of both the tallow and train oil by degras had the opposite effect on water absorption, again without changing the strength or other properties. (4) Replacement of 40 and 70% of the train oil by mineral oil increased the water uptake by 16 and 29% respectively, without changing any other properties appreciably. (5) Replacement of 40 and 70% of the tallow by paraffin had no appreciable effect on any measured property. (6) Addition to the fat-liquor of 0.5 and 2.0% of Smenol WO Concd. (a sulfated fat alcohol product) increased the values obtained in the strength tests by 5-15 and 10-23% respectively without increasing the % stretch at break. Water absorption (2 hrs.) increased by 27 and 64%, and water absorption (24 hrs.) by 5 and 18%. Wetting time decreased by 9 and 16%. The air transmission coefficient increased by 5 and 10%, but the water vapor transmission coefficient was not changed. Thermal conductivity decreased by 3 and 7%. The increased strength is laid to better pentration of fat into the fibers. *Chrome leather:* (1) The physical properties of standard leathers fat-liquored with castor oil of low and high degree of sulfation were practically identical. The former leather contained extractable fat: bound fat = 3.1 and the latter about 3.2. Changes made in the amount and composition of fat liquor had the same effect, or lack of effect, regardless of degree of sulfation of the castor oil. (2) A 40% reduction in amount of fat-liquor given reduced the strength and % stretch at break by only 4-9%, and a 40% increase in given fat-liquor increased the strength by 4-8%. Wetting time was decreased 5-8% by the reduction, and increased 5-7% by the increase in fat-liquor, but water uptake (2 and 24 hrs.) was not appreciably affected. No other properties were much affected. (3) Replacement of 50% of the sulfated oil by neutral castor oil, or train oil, increased the strength by 4-12% and 7-18% respectively; but

replacement of 25% or 50% of the sulfated oil by mineral oil had no effect on the strength. The wetting time was increased by all these substitutions, especially by 50% mineral oil (17-35%) but water uptake was reduced appreciably only by 50% mineral oil (10%). The air transmission coefficient was lowered by 8-15% by introduction of neutral castor oil or train oil, but not by mineral oil, and the water vapor transmission coefficient was unaltered. The fact that only very slight (barely significant) changes in results of strength tests are produced by rather drastic changes in amount and composition of oil applied is noteworthy. The stitch tear test appears to be more sensitive to changes in amount and kind of fat than either the tensile strength or the slit tear test. H. B. M.

Influence of Drying and Mechanical Finishing Operations on the Properties of Shoe Upper Leather. F. Stather and H. Herfeld. *Ges. Abhandl. deut. Lederinsts. Freiberg/Sa.* No. 4, 84 (1950). See preceding abstract. The normally stuffed vegetable upper leathers, and the normally fat-liquored chrome upper leathers described in the previous article were subjected to the same physical tests as before, with variation in drying conditions and mechanical operations. The physical properties of vegetable leather dried 4 days at 20°C. and 75% relative humidity (R. H.) were not different from those of leather dried 1 day at 40° and 75% R.H. and 1 day at 40° and 50% R.H. (standard conditions), but the strength of vegetable leather dried 1 day at 60° and 75% R.H. was 10-13% lower than the standard. Elongation at break was not affected. The leather dried at 60° absorbed 20% less water (24 hrs.), and showed about the same decline in transmittance of air and water vapor. Varying the relative humidity during drying from 30 to 90% at 40° had no detectable effect on the physical properties. No change in any physical property of chrome upper leather could be detected when the temp. of drying was varied from 20 to 60°C. (24 hrs., 50% R.H.), or when the R.H. during drying was varied from 25 to 75% (40°, 24 hrs.). The strength of vegetable upper leather was unaffected by whitening and by graining, but the strength per unit thickness increased with the pressure applied at glassing, owing to diminution of thickness. Whitening and graining increased the rate of water absorption (but not % water absorbed in 24 hrs.); both rate of water absorption and % absorbed in 24 hrs. decreased with increasing glassing pressure. Air transmittance coefficient was increased by whitening and graining; and decreased with increasing glassing pressure. Water vapor transmittance coefficient was unaffected by whitening, decreased with increasing glassing pressure, and was increased by graining. Thermal conductivity was increased by glassing and decreased by graining. No difference in physical properties of chrome leather could be detected when time of sammying in sawdust was increased from 12 to 24 hrs. The only detectable effect of increasing tension when the leather was tacked was a slight decrease in % stretch at break. Increasing the ironing pressure from 60 to 200 atmospheres gave a considerable increase in strength (about 10%) on the thickness basis (owing to diminution of thickness), a decrease in elongation at break, slight decreases in water uptake (rate and amount) and in water vapor transmittance, a marked decrease (25%) in air transmittance coefficient, and a marked increase (20%) in thermal conductivity. Raising the ironing temp. from 40 to 80°C. had no effect on any property. The effect of glazing, especially the kind of glaze finish, is to be treated in a separate article. H. B. M.

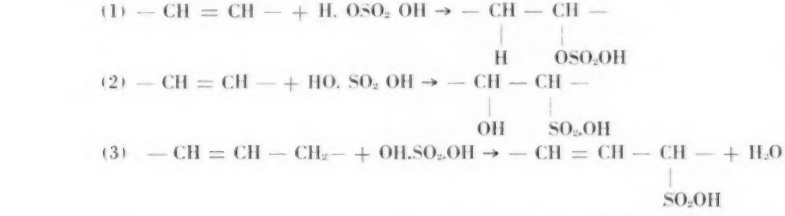
Influence and Kind and Amount of the Solvent Employed on the Properties of Leather Creams. H. Herfeld and W. Kalzenan. *Ges. Abhandl. deut. Lederinsts. Freiberg/Sa.* No. 3, 79 (1950). This work was done with creams of known compn. prepared in the laboratory. The base for non-aqueous creams, and for creams containing both water and organic solvent, was a blend of I.G. Wax 14, hard ceresine 46, soft ceresine 12,

and paraffin 28%. For water creams the base contained I.G. Wax 44, ceresine 11, "emulsion wax" (a paraffin oxidation product) 44, and K_2CO_3 for saponification 1-5%. The variables studied were ratio of total solvent to waxes, ratio of turpentine to benzine, and ratio of water to organic solvents. Properties studied were consistency, ease of filling into containers, change of consistency with temperature, rate of loss of solvent by evaporation, corrosion of containers (iron and zinc), ease of application to leather, penetration of wax into leather, surface accumulation of wax on repeated polishing, luster, ease of polishing, retention of luster after exposure to water, and restoration of luster by repolishing, and the degree of water repellency imparted to leather. In general, all properties of the film improve with increasing ratio of solvent to wax—the practical limit being the ratio that gives the desired consistency for boxed or tube creams. Partial replacement of turpentine by benzine results in higher luster, easier application, and less corrosion, but gives a harder cream showing enhanced solvent loss by evaporation, less penetration, more accumulation of surface wax (because benzine does not cut the old wax), and lower water repellency. Partial replacement of turpentine by benzine is feasible, and economical, but then the composition of the base should be adjusted. Increasing the ratio of water to organic solvents results in less variation of consistency with temperature above 0°C., but all properties of the film are progressively impaired. The mixed-type cream should contain at least 20% of organic solvent. The pH value of mixed-type and water-type creams need not, and should not, exceed 8. H. B. M.

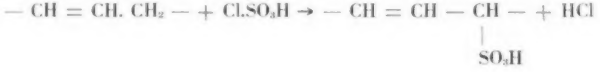
General Meeting and Conference. *J. Soc. Leather Trades' Chemists* **36,** 297 (1952). In addition to the business meeting the following papers were presented: "A Shoeman's Views on Soling Materials." "The Intrinsic Properties Which Make Leather So Unique." "Surface Films—A New Approach to the Mechanism of Tanning Reactions." "The Reactivity of Protein Fibers: Some Analogies Between Keratin and Collagen." "Some Applications of Microchemistry." "Modern Views on the Architecture of the Collagen Fibrils in Pelt." R. H. T.

The Constitution of Sulfated Oils III. The Constitution of Commercial "Ricinoleic Acid" and a Study of Its Reaction with Sulfuric Acid. By D. Burton and L. F. Byrne. *J. Soc. Leather Trades' Chemists* **36,** 309 (1952). Ricinoleic acid is said to polymerize on standing. The commercial acid may contain ricinoleic acid, its lactone, its lactide, the estolide, monobasic di-ricinoleic acid, and dibasic di-ricinoleic acid. It may also contain l-ricinoleic acid as well as the dextro isomer. Analysis of the sample under study indicated that it contained little of the poly-ricinoleic acid but considerable lactone. Calculactions, based on the analytical data and on the assumption that there was no significant amount of estolide or dibasic di-ricinoleic acid present, showed the following analysis: ricinoleic acid 61.4 per cent; lactone 26.8 per cent; lactide 11.3 per cent. Because of the presence of the hydroxyl group in ricinoleic alid, sulfation may take place through simple esterification of this group with sulfuric acid. There is evidence in the literature that this takes place to the exclusion of the reaction at the double bond, when the reaction is carried out at temperatures below zero, until the esterification reaction is completed. Thereafter the reaction slowly takes place at the double bond. Since sulfation is usually done at higher temperatures, for this study four sulfations were made at higher temperatures. Oil I was made with 25 per cent of the oil weight as concentrated sulfuric acid at 30 degrees C. for 4 hours; oil II with 40 per cent of concentrated acid at 30 degrees for 6 hours; oil III with 37.5 per cent concentrated acid at 50 degrees for 6 hours and oil IV with 39 per cent chlorosulfonic acid plus 20 per cent glacial acetic acid at 30 degrees for 4 hours. Details of all sulfations are given. A table gives the g. equivalents of sulfated products yielded by

100 g. equivalents of ricinoleic acid. A drop in iodine value for oils I, II and III shows clearly that sulfation of ricinoleic acid is not a simple case of esterification of the hydroxyl group. The largest iodine value drop is in oil II. Oils I and III have about the same drop, which is just a little over half that of oil II. Oil IV has practically no iodine drop but a greater content of sulfate and sulfonate, although all four oils show considerable of both. In the first three oils the following reactions are indicated:



The third reaction takes place in part in oil III made at 50 degrees. The relative amount of sulfonate to alkyl sulfate is increased by increasing the proportion of acid from 25 to 40 per cent and to a much greater extent by raising the temperature from 30 to 50 degrees C. Since there was practically no drop in iodine value in oil IV and yet considerable sulfonate formed it is evident that there was a reaction other than the esterification of the hydroxyl. Since the double bond was preserved it is probable that the reaction is as follows:



R. H. T.

The Separation of the Tannins and Other Constituents of Myrobalans. By R. S. Asquith. *J. Soc. Leather Trades' Chemists* **36,** 316 (1952). The myrobalan powder was first extracted with acetone to give a soluble infusion which was fractionated by extraction with ethyl acetate at pH 7 and then at decreasing pH values. Three main fractions were obtained: (1) the fraction extractable at pH 7 pH 6, (2) the fraction extractable below pH 3 and (3) the residual fraction insoluble in the ethyl acetate. The acetate fractions were evaporated to small volume and the material precipitated by addition of petroleum ether. The (1) fraction showed gallic acid probably due to decomposition. After removal of the gallic acid a definite chromatogram of digalloyl glucose was obtained. There was also glucose but no ketose. Hydrolyzed with water, by heating in a sealed tube over night, gallic acid and pyrogallol were found. At least two other constituents, other than digalloyl glucose and its decomposition products were separated but could not be identified. From analyses and chromatograms of water hydrolysis products, it is considered that there may be mono-galloyl-mono m-digalloyl glucose present. Examination of fraction (2) indicated that it contained all the acidic compounds present in the acetone infusion. Chromatograms showed at least three substances which could not be identified even when separated into four subfractions by use of a silica partition column. As no sugars were detected and gallic acid and pyrogallol were found upon hydrolysis, it is thought this fraction may result from the splitting off of digalloyl glucose from chebulinic acid. In this fraction there were also found split acid and an unidentified compound yielding split acid and gallic acid on hydrolysis. Fraction (3) showed two unidentified compounds. It was very stable but yielded to prolonged hydrolysis with water giving gallic acid, split acid and ellagic acid. R. H. T.

Effect of Tanning in Protecting Collagen Against Enzyme Digestion. By F. G. Lennox and H. M. Forss. *J. Soc. Leather Trades' Chemists* **36,** 322 (1952). Since measurement of putrefactive action of bacteria directly is difficult and the putrefaction is due to the actions of enzymes secreted by the bacteria, a study of the effects of enzymes on protein was undertaken. It was found that papain in the presence of urea is an effective digestive agent. Collagen was prepared by depickling sheepskin, which has been pickled in sulfuric acid and sodium chloride, with sodium chloride and sodium acetate until neutral. It was then dehydrated and degreased with acetone. Tannages were vegetable, formaldehyde, chrome, alum and syntan. Digestion was carried out for 50 min. at 50 degrees C. and 75 min. at 35 degrees C. A solution containing 1 per cent papain, 0.1 M sodium bisulfite and 3 M urea at pH 8 was found to give the best digestion. Tanning procedures are described. Enzyme digestion, shrinkage temperature and alkali solubility were measured. Methods of measuring are described. The following table gives the results obtained:

Tannage	Enzyme Digestion at pH 8%.	Shrinkage Temp. °C.	Alkali Solubility %	
Chrome	2	100	29	
Formaldehyde	11	84	8	50 min. at 50 °C.
Vegetable	25	78	34	
Syntan	23	72	79	
Alum	38	54	67	75 min. at 35 °C.
Untanned	85	59	85	

It may be noted that, according to shrinkage temperature, the alum tannage is very poor, whereas the enzyme digestibility shows good tannage. Since usage has shown alum tannage to be effective, it may be concluded that the enzyme digestibility is the better means of assessing the tanning value. R. H. T.

Vegetable Tanning VII. The Effects of Salts of Weak Acids. By D. Burton, J. M. Harrison and T. Turner. *J. Soc. Leather Trades' Chemists,* **36,** 342 (1952). Sodium formate was chosen as the weak acid salt. Two series were run. In the first 50° Bk. liquor was brought to pH 3.5 with formic acid and diluted to 40 degrees. In the second series the same 40° Bk. liquor was used but the pH was adjusted to 3.5 after each formate addition. In each series there were additions of sodium formate of 0, 20, 60, 100 and 300 mg. equiv. per liter. Pelt pieces were cut to get adjacent comparison pieces in all liquors. Examination was made after 3 days and some were taken out at 10 days and the balance at 34 days of tannage, which was carried out at room temperature. In general the firmness of the wet leather is decreased by the salt additions. No effect is seen on color when the pH is allowed to rise with the salt additions. When the pH is maintained at 3.5, color darkens with increased salt additions up to 100 mg. equiv.; but the 300 mg. equiv. gives a lighter, brighter color than when there has been no salt addition. Comparing results with those obtained in previous studies using sodium chloride and sodium sulfate it is noted that: (a) sodium formate does not increase the speed of penetration of tanning matters except when 300 mg. equiv. per liter are present, while the chloride and sulfate cause marked acceleration at all concentrations: (b) the addition of any of these salts causes the liquor to contain more salts of weak acid after the pelt has been tanned in it; (c) all these salts decrease the degree of tannage and the tanning figure, the extent depending upon the quantity added except in the case of the second sodium formate series, where the degree of tannage increases up to 60 mg. equiv. per liter and then decreases; (d) these salts all increase shrink-

age temperature slightly again excepting the second sodium formate series; (e) when the liquor contains salts of strong acids in addition to the weak acids present, the pelt takes up strong acids and thus shows a positive Procter-Searle value. R. H. T.

The Combination of Cation Active Compounds with Collagen and Chrome Tanned Collagen. By D. A. Plant. *J. Soc. Leather Trades' Chemists* **36,** 351 (1952). The difficulties encountered in determining the protein groups involved in chrome tanning are discussed briefly. For this study the quaternary ammonium salt cetyl trimethyl ammonium bromide was selected. Comparisons were made between hide powder and chrome tanned hide powder treated with 2 m. equiv. of the quaternary salt per g. hide powder protein in O.1/N potassium chloride. The solutions were adjusted in pH with HCl and KOH; and M. equiv. of the quaternary salt combined per gram of dry collagen plotted as a function of pH from 2 to 12. The hide powder gives a curve similar to an alkali combination curve but the amounts combined are greater, indicating that the combination of hide powder and the quaternary ammonium salt are not stoichiometric. This curve is sigmoid between pH 2 and 8. The chrome tanned hide powder shows no such sigmoid curve between pH 2 and 8, the hump between 5 and 8 being absent. This indicates that the free or non-zwitterion groups are blocked by the chrome tannage. The uptake of the quaternary salt at pH 7 (in the free carboxyl titration region) and at pH 12 (in the zwitterion back titration region) was determined as a function of cation concentration. Both showed increased combination with increased cation concentration, more at pH 12 than at 7. At the latter pH an adsorption maximum was reached. There was no evidence of a two-stage adsorption process. Dilution of the quaternary salt, maintaining the same amount of the salt in increasing amounts of solution, did not alter the uptake up to fifty fold dilution. Calculations of chrome uptake by collagen, based on the assumption that only the free carboxyls are involved, gives 1.81 per cent Cr or 2.63 per cent Cr_2O_3. This may be considered the minimum fixation necessary to give cross linkage of the polypeptide chains and hence a rise in shrinkage temperature. The higher fixation actually encountered in practice may be due to fixation of complexes containing more than 2 Cr atoms, used in the calculation, or to chrome-chrome fixation not involving further reaction with reactive groups of the collagen. It was not possible to determine to what extent zwitterion carboxyl groups are involved due to lack of stoichiometry in the combination between cetyl trimethyl ammonium bromide and the collagen at pH values above 9 where back titration of the zwitterions commences. R. H. T.

The Degreasing of Woolskins. By R. F. Innes and K. G. A. Pankhurst. *J. Soc. Leather Trades Chemists* **36,** 358 (1952). The object of the investigation was to see whether degreasing, as carried out with sheepskins, would cause objectionable felting with woolskins and whether, if so, a mild degreasing treatment would be advantageous. Two tests were made on sun-dried Australian woolskins. In the first the skins were paddled in a mixture of 50 lb. of Corilene D.G. (an alkyl phenol-ethyleneoxide condensate) in 100 gal. of water at 35°C. and 17 r.p.m. for 105 min. In the second the skins were hydro-extracted for 5 min. and then sponged on the flesh side with Corilene D.G. (½ gal. used on 10 skins) and left flesh to flesh over night. On the basis of skins judged greasy, fair and non-greasy, the first method showed 45 per cent efficiency and the second 75. In both there was decrease in grease in the skin but some increase in grease in the wool. As the sun-dried Australian skins are not so greasy as the South American salt-dried skins, tests were also made on the latter. The prepared skins were divided into 3 randomized packs. The first was treated on the flesh side with neat Corilene D.G. and laid flesh to flesh over night. The

second was similarly treated except that an equi-volume mixture of Corilene D.G. and Pool burning paraffine was used. The third was untreated for control. All were water washed in the morning. The first showed 10 per cent greasy skins, the second 12 per cent and the control 31 per cent. Both degreasing methods have been effective with little difference between them. In another experiment sun-dried Australian skins were slow drummed (3 r.p.m.) in Pool burning paraffin containing 5 lb. Corilene D.G. per 200 gal. The pack weighed 264 pounds. Drumming was carried on at about 38 degrees for 3 hrs. No felting occurred. A similar experiment was carried out with dry-salted South American skins fast drummed (12 r.p.m.). At 2 hrs. 20 min. felting appeared so drumming was discontinued. In the first test there was little evidence of effective degreasing although analyses showed that some grease had been removed. The second showed effective degreasing. The experiments suggest that a useful degree of degreasing can be achieved with woolskins by applying the normal degreasing process as worked out for pickled sheepskins and that felting does not become serious up to 2 hrs. and 20 min. drumming at 12 r.p.m. Encouraging results are also obtained when the skins are dry pickled and then painted with an equi-volume mixture of Corilene D.G. and paraffin, piled flesh to flesh over night and rinsed in a paddle of warm water in the morning. Both these processes are improved if the skins are hydro-extracted after degreasing. R. H. T.

Note on the Uptake of Grease During the Fatliquoring of Vegetable Tanned Sheepskins. By K. G. A. Pankhurst. *J. Soc. Leather Trades' Chemists* **36,** 363 (1952). The object of the work was to determine how the uptake of grease from a fatliquor was dependent upon the grease content already present. Four vegetable tanned basils were laid out in 2 in. squares, with 1 in. margins between squares, both sides being symetrically marked with relation to the backbone. Alternate squares were cut out and the grease content determined by extraction with petroleum ether. The skins were then fatliquored with sulfated castor oil in the usual manner. After drying the grease content of the remaining squares was determined. The results showed that, as long as the grease content of the hide was below 30 per cent, grease was taken up by the pelt; but, if the grease was over 30 per cent, degreasing took place. In other words, the sulfated castor oil degreased the high grease areas and added grease to the low grease areas, thus generally levelling the grease content. The author feels justified in expressing it as a quadratic equation $G = A + BG_o - CG_o^2$ in which G_o is the original grease content and G the grease content after fatliquoring with sulfated castor oil. R. H. T.

Kubelka Water Absorption and Free Water. Determinations on Vegetable Tanned Sole Leather. By L. M. Hill. *J. Soc. Leather Trades' Chemists* **36,** 365 (1952). Water adsorbing capacity is important in the evaluation of sole leather. Free water is considered to be the volume of water required to fill free space present in finished leather plus the amount required to satisfy the residual swelling capacity of the fibers. Determination of free water requires at least 72 hrs. The Kubelka water absorption requires only 18-20 hrs. The free water test is a recognized guide to quality. The object of this work was to determine whether the Kubelka test could be satisfactorily substituted, to study the effect of sampling from the fore-end corner of the bend and to arrive at a Kubelka water absorption figure which would be regarded as a reasonable maximum upper limit for assessing South African vegetable tanned leather for outersoles. The position of the belly-shoulder, fore-end corner is known to be the position where water absorption is the highest. Hence a seven-inch square was taken from this point and from it seven 2-inch squares were cut, three along the top, three along the bottom and one at the shoulder edge in the center. The

balance of the piece was used for hide substance and moisture determinations. All samples were dried, then conditioned by holding at 65 per cent R.H. and 70°F. for at least 48 hrs. The Kubelka apparatus is described and method of determination given. Kubelka water absorption equals the decrease in water volume measured in the instrument times 100 divided by the original conditioned weight. The saturated leather sample from the Kubelka determination was rinsed lightly, blotted and weighed saturated (W_t). It was then dried (W_d) and finally reconditioned to the original (W_o). The free water equals $\frac{W_t - W_d}{W_o} \times 100$.

Twenty-two bends from different sources were sampled and determinations made on the seven pieces from each sample. There was little variation between the seven pieces. In general the free water was higher than the Kubelka water. The former varied from 39.9 to 59.4 per cent and the latter from 33.3 to 58. While the figures are not interchangeable they are closely associated. The conclusion is that a maximum Kubelka value of 55-58 per cent on a sample cut from the belly-shoulder, fore-end corner of the bend will eliminate belly leather, most shoulder leather and poorly tanned leather. Most determinations would fall well below this figure. R. H. T.

The Influence of Fatliquor on Spue Formation and Run in Gloving Leather. By K. G. A. Pankhurst, R. G. Mitton, R. F. Innes and R. N. Johnson. *J. Soc. Leather Chemists* **36,** 379 (1952). Fatty spue may be due to natural fat or fat introduced through fatliquoring. If concentrated in the neck, tail and backbone regions and at the juncture of limbs, it is probably natural fat. If well distributed all over the pelt, it is probably from fatliquoring. Spue from natural fat may be eliminated by degreasing before tanning; but spue from fatliquor is not so easily remedied or avoided. To overcome fatliquor spue it would seem necessary to first discover the properties of the oil causing it. The object of this experiment was to try to associate spue formation and run with the physical and chemical properties of the oils which might be used. Nine oils were selected and specially sulfated. Bursoline SCO, believed mainly sulfated herring oil, was added as a control. The oils used were herring, low sulfated seal, high sulfated seal, cod, whale, sperm, neatsfoot, oleine and palm kernel. The significant physical and chemical properties were determined. These included combined SO_3 on total fat, iodine value, sapon. value, melting points of neutral oil and free fatty acid, melting point of the sulfated oil and relative viscosities of the sulfated and unsulfated oils. The different oils were applied as fatliquor in three different tanneries and their effect on spue formation and run studied. Examination within a month, with temperature at 40-50°F., showed the following grading going from no spue in group (a) to the most in group (d). Group (a) oleine and palm kernal, (b) low sulfated seal, cod and herring, (c) high sulfated seal, whale and sperm, (d) neatsfoot. At six weeks, temperature 60°F., they showed less over-all spue, (a) still being spue free and (d) showing the most. Groups (b) and (c) could not be differentiated at this stage. Approximately a year after finishing no spue was detectable at temperature between 35 and 60°F. Run classification, going from most run to least, is as follows: sperm, high sulfation seal, cod, whale, low sulfation seal, herring, palm kernel, neatsfoot, oleine. While this should be regarded as preliminary, the best over-all results were obtained with sulfated cod oil. R. H. T.

Note on the Neutral Salt Effect in Chrome Tanning. By S. G. Shuttleworth. *J. Soc. Leather Trades' Chemists* **36,** 390 (1952). The author has criticized Gustavson's theory that "the retarding and decreasing effect of neutral sulfates on the chrome fixation by col-

lagen from solutions of basic chromium sulfate is mainly due to the conversion of electropositive chromium into uncharged complexes, which possess a lower degree of affinity for collagen." The criticism is based upon the effects of heat and time, neutral salt effect on other chromium complexes spectrophotometric evidence, benzidine hydrochloride analyses and Gustavson's interpretation of his own results. The differences in terminology encountered are briefly discussed. R. H. T.

Improved Degradation Methods for Examination of Wattle and Allied Tannins. By D. G. Roux. *J. Soc. Leather Trades' Chemists* **36,** 393 (1952). Abnormally low yields of significant end products derived from the degradation of black wattle tannin, as obtained in past studies, indicate the value of improved methods. The methods studied in this work were the alkali fusion of the purified tannin and the alkaline permanganate oxidation of the methylated tannin. For the former the extract was purified by alcohol and lead salt precipitation and extraction with ether. The purified tannin was fused in a steel bomb, after being intimately mixed with a 6:4 eutectic mixture of KOH:NaOH, and held at 200°C. for one hour. By this treatment an ether soluble phenol and phenolic acid fraction of 43 per cent yield was obtained. From this the yields, compared with previous yields, were as follows, the yield by the present method being given first in each case: Beta resorcyclic acid 4.2 per cent and none. Gallic acid 11 per cent and less than one. Protocatechuic acid a little and none. Resorcinol 10.5 and one. Pyrogallol less than one per cent and none. Phloroglucinol a trace and none. Obviously those in which none had been found before were isolated for the first time. For the alkaline permanganate oxidation 5 g. of lead salt purified tannin (methylated with dimethyl sulfate) of maximum methoxyl content (36.5 per cent) in 600 ml. of acetone and water mixture 1:1 was refluxed with the slow addition of potassium permanganate. A total of 65 g. was added in 5 g. quantities each time the permanganate color was discharged. The managanese dioxide was filtered out and washed with a fresh acetone water mixture. Filtrate and washing were concentrated and acidified, then extracted with ether. After drying and removal of ether, the residue was vacuum sublimed at 140-180°C. There was recovered a red acidic residue from the sublimation, white sublimates and a certain amount of unoxidized material from the original oxidation. From these there was a yield of at least 20 per cent of o-trimethyl gallic acid and veratric acid mixture. The significance of some of the degradation products and the similar behavior of tannins and catechins during degradation are discussed. R. H. T.

An Apparatus for the Simultaneous Determination of Grease in Large Numbers of Samples of Leather. By S. C. Ellis, R. G. Mitton and K. G. A. Pankhurst. *J. Soc. Leather Trades' Chemists* **36,** 401 (1952). Determination of grease by individual Soxhlet extraction is too time-consuming when a large number of determinations are required. For 1000 determinations, using six Soxhlet extractors, practically 80 days with continuous attention is necessary. The apparatus and method described here will give the 1000 determinations in about 25 days with only 4 or 5 days continuous attention. The apparatus makes use of solvent extraction; but the samples are not ground and are extracted as is. The grease is determined by taking the weight of the leather before and after extraction. A large extractor, which is described, is used and many pieces of leather may be extracted at the same time. To avoid loss of weight due to small fibrous pieces becoming detached during the extraction the edges are carefully trimmed when preparing the samples. It is necessary that the leather be uniformly conditioned before and after degreasing. First the samples are dried for 24 hrs. at 45°C. They are then suspended in a current of air under controlled temperature and humidity until they reach a point where they vary in weight, both

up and down, within acceptable limits. The samples are then all degreased together and again dried at 45°C. for 24 hrs. and again conditioned to uniformity under similar controlled temperature and humidity before weighing as grease-free leather. Control samples of similar leather are put through the same routine with exception of the actual degreasing. Let w_1 and w_2 be the weights before and after degreasing and c_1 and c_2 be the original and final weights of the control sample. Then the grease removed is $w_1-(w_2c_1/c_2$. Tests made showed that the results obtained are similar but slightly higher than those obtained with Soxhlet extractors. This demonstrates that the results are well within the required accuracy for most work of this kind. R. H. T.

Analytical Studies of Tannin Content of the Bark of the Turkish Pines, Pinus nigra, Pinus silvestris, Pinus pinea and Pinus brutia. By O. Gerngross and E. Gülbaran. *Das Leder* **3,** 169 (1952). The outer, weathered, gray bark of *Pinas brutia* was found to contain less tannin (13-19%) than the inner red bark (17.4-24.9%), therefore only the deep red, inner bark was used. All tannin analyses were made by the shake method with freshly chromed hide powder. *Pinus nigra*, black pine, is the most common pine in Turkey. Tannin content of the bark was low. It varied from 2.7 to 5.4 per cent. *P. silvestris*, scotch pine, is widespread in central Europe and Turkey. Tannin content of the bark decreased from an average of 10.8 per cent in trees 20 years old to an average of 3.0 per cent in trees 255 years old. Variation of tannin content with height of sample above ground was irregular, probably because of improper sampling. Purity varied from approximately 42 to 62. *P. pinea* is valued for its edible nuts. The bark comprises 16-18% of the volume of the trunk. Tannin in the bark varied from 12.8 to 20.2 per cent, and purity from 74 to 83. The pH value of the analytical extract was 3.4 to 3.8. Age of the tree had no influence on tannin content of the bark. The large (250 grams) cones are collected for the seeds, but the waste cones contain only 3.3 per cent tannin. The average tannin content of *P. brutia* trees 20 years old, was 12.8 per cent (8.8-17.8) and of trees 225 years old, 27.9 per cent (21.9-34.1). The pH value of the extract was 3.6-4.5. There was a slight decrease in tannin content with height of sample above ground. I. D. C.

Comparative Determiation of Tan and Combining Values of Vegetable and Synthetic Tanning Materials and Cellulose Extracts. By Oskar A. Muller. *Das Leder* **3,** 174 (1952); cf. *This Jour.* **47,** 299. Ten different extracts were investigated by the standard method, with careful control of amount of tannin, concentration and acidity of the liquor, temperature during tanning and washing, and quality of the skin. Three types of skin material were used: powder, shavings and pieces. They were made from calfskin that had been brought to pH 4-4.5 with acetic acid, dried with alcohol-acetone and that had an ash content of not more than 0.1 per cent. The extract solutions, containing 4 per cent tannin, were filtered through paper until clear. Vegetable extracts were used at their natural pH; other extracts were brought to pH 3.5 (glass electrode) with sulfuric acid or sodium hydroxide. The extract solution was added in portions of 0.1, 0.2, 0.3 and 0.4 of the total at intervals of 0, 2, 8 and 24 hours. The skin was presoaked in the proper amount of water, including skin moisture, so that the tannin concentration in the liquor was 3 per cent. During tanning the pH value was maintained at as near 3.5 as possible by addition of acid at intervals of 1-2 hours. A correction of tan value (GW) for the volume of acid was necessary. Four different times of tanning were used for each extract. These were 3, 10, 21 and 42 days. Washing of the tanned hide for determining combined tannin was for 15-20 hours with liters of water. The washing was repeated for all extracts, except cellulose extract. Vegetable extracts gave the highest and cellulose extracts the lowest tan

values. Powder gave the highest tan values and pieces the lowest in all tests, including the 42-day tannage. This difference could not be due to diffusion differences but rather to setting free of tanning active collagen groups during grinding to a powder. For vegetable extracts the combining values were higher with pieces than with powder, but the reverse was true for syntans and cellulose extracts. Increasing the amount of tannin in the liquor from 200 to 300 per cent increased tan and combining values of vegetable extracts appreciably, but had less effect on syntans and cellulose extracts. With the last, 100 per cent tannin was almost sufficient to complete the tanning. The following values were found with pieces of hide, using sufficient time and excess of tannin:

	tan value	*combining value*
Vegetable extract	100-110	80-90
Syntans	60-80	60-70
Cellulose extract	50	50

The matter lost during washing was greater with hide powder than with hide pieces. With vegetable extracts the loss decreased only slightly as time of tanning was increased. The loss with cellulose extracts was extremely small. I. D. C.

The Tannin Balance in Sole Leather Tanning. By J. Wagner. *Das Leder* **3,** 178 (1952). The tannin lost during sole leather tanning is the difference between total tannin put into the process and the sum of the tannin in the liquors, in leather in process and in the finished leather. Estimates of this loss have varied from 13% to 38%. The loss is influenced by the following factors: 1, kind of tannage (vat, combination, rapid); 2, degree of deliming; 3, tanning time; 4, tannin concentration; 5, kind of tanning material; 6, acidity of the tail liquors; 7, temperature; 8, bark leaching yield; 9, system of discharging spent liquors; 10, transportation losses; 11, amount of tannin in sludge; 12, manner of washing and setting out the leather; 13, kind and amount of leather waste from shaving and buffing; 14, technical organization of the tannery; 15, method of sampling for control analyses; 16, method for determining tannin (filter or shake). Fiksl (Inaug. Dissertation, Brünn 1930) lists the tannin losses as follows: Chemical changes in the liquors, 12.73%; sludge from dilution of liquors, 2.50%; sludge from action of acid, 0.76%; sludge caused by water hardness, 2.83%; wash waters, 0.90%; waste liquors, 4.71%; leather buffings, 0.73%; analytical variations, 2.45%. The last loss results from variations in sampling (sample location, etc.) and in analysis. I. D. C.

Quality Requirements of Leather for the Shoe Industry. (Comments on the Lecture of G. Rheinberger). By K. Eitel. *Das Leder* **3,** 265 (1952). Most of the requirements outlined by Rheinberger *(Das Leder* **3,** 247) can be met, but some price adjustments would be necessary. More cooperation between the shoe manufacturers and the leather industry would be of value. I. D. C.

The Formation of Drawn Grain During Vegetable Tanning. By E. Quendt. *Das Leder* **3,** 269 (1952). Drawn grain is of two types. In one type, "milled grain", the entire leather surface is covered uniformly with wrinkles and somewhat resembles cobblestone paving. It is caused entirely by mechanical movement of the hides in the drum during tanning. The other type, "shrunken grain", consists of long parallel furrows, and is the

result of acid swelling. If a hide in the fallen condition is partly tanned so that the grain layer, but not the interior, is tanned, and it is then put in an acid liquor, the inner layers will swell and contract, causing wrinkling of the grain layer. In practice, this may occur when a fallen hide is put into acid liquors containing enough astringent tannin to set the grain (not just to color it). Acid will penetrate ahead of the tannin and swell the middle layers causing drawn grain. A pickled skin may show drawn grain in a vegetable tan liquor if the liquor does not contain enough salt. Salt will diffuse from the skin to the liquor and the middle skin layers will then swell and cause grain wrinkling. If the hide is swollen when tanning starts, it will not show drawn grain if it is transferred later to less acid liquors. I. D. C.

A Wood Boring Beetle in Spruce Bark. By T. Stirtz. *Das Leder* **3,** 273 (1952). Spruce bark stored in a tannery in the Black Forest was found to be badly damaged by a wood boring beetle, *Stephanopachys substriatus Payk.* of the Bostrychidae family. The dark brown, cylindrical beetles are 4-6 millimeters long. They do considerable damage to commercial timber in the tropics. Although considered at first a new type of damage, a note *(Das Leder* **3,** 303) points out that damage to tan bark by this beetle has been reported by Hausam and Liebscher [*Collegium* No. **875,** 65 (1943)] and elsewhere. I. D. C.

Process for Bleaching Vegetable Tanned Leather. By W. Grassmann and P. Stadler. *Das Leder* **3,** 289 (1952). Bleaching leather with acid and alkali is hazardous so that there is an increasing tendency to change to tanning sulfo acids or tanning oils. The last do not bleach in the same manner as pure acids, but remove surface tannin from combination with hide substance. They are good if used immediately after tanning and before drying the leather, but are not effective after the leather has dried and a surface deposit of tannin has formed and oxidized. It has long been known that organic solvents and especially their mixtures with water, have a detanning action. A combination of acid and acetone was found to have a much greater detanning action than either acid or acetone alone. A maximum effect was found with from 30 to 60 per cent of acetone. In 30 per cent acetone, even 0.1 per cent oxalic acid in the solution gave pronounced bleaching, whereas a much higher concentration of acid without acetone did not bleach satisfactorily. More surprising was the fact that, in the presence of acetone, there was good bleaching action even when a large proportion of the oxalic acid had been neutralized. In 30 per cent acetone half of the acid could be neutralized, with consequent increase in pH value from 1.9 to 3.7, with no loss of bleaching action. Many organic and inorganic acids were effective, but whether there are quantitative differences between them has not been established. Auxiliary syntans were found to be as effective as acids for bleaching. Those tried were Basyntan FC, FCBJ, Tanicor HSB, HZ, HN, HS (powd.) HSK, Tanigan BLG (powd.) and Irgatan FL. A considerable proportion of the syntan could be neutralized. Without acetone, syntans had only slight bleaching action and, if partly neutralized, were entirely unsatisfactory. In general, bleaching was appreciable in 15 minutes and reached a maximum in 30 minutes. A search was made for cheaper and better solvents than acetone. Suitable solvents were very soluble in water and had low boiling points. Use of solvents with boiling points as high or higher than that of water, such as various glycols, resulted in slow drying of the leather and migration of tannin to the leather surface. Such treatment sometimes resulted in darkening rather than bleaching of the leather. There was a specific solvent effect. Acetone, tetrahydrofurane or a methylacetate methyl alcohol mixture were suitable for bleaching but methyl or ethyl alcohol were without effect. The first group are proton acceptors and the last group, proton donors. A tentative suggestion is made that solvents

of the first group may form hydrogen bonds with the tannins and thus remove them from combination with the protein. The role of water, however, must be clarified. I. D. C.

The Existence of Chloro-hydroxo-complexes in Basic Chromium Chloride Solutions. By K. H. Gustavson. *Das Leder* **3,** 293 (1952). The existence of chlorochromium complexes has been questioned by Küntzel Erdmann and Sparkas (this *jour.* **47,** 799) based on conductivity measurements in solutions containing 1 gram of chromium per liter. The chloro group has only a small complex stability, so that formation of chlorohydroxochromium complexes depends on a sufficiently high concentration of chromium and chloride ions. Gustavson has therefore used solutions having a high chromium content, containing at least one mol. of sodium chloride per chromium atom, and has made measurements either on the concentrated solutions or immediately following a suitable dilution. There is an equilibrium between coordinately and ionically bound chlorine, therefore complex bound chlorine must exist in dilute solutions, although the amount may be too small to be detected by the usual methods, if the basicity is low (less than 50). In highly basic chromium chloride solutions (over 66%) the amount of uncharged chlorochromium complex is independent of the concentration of chromium and chloride. Previous work which indicates the formation of chloro complexes in basic chromium chloride solutions is reviewed. This work includes investigations with ion exchangers of both the cationic and the anionic types, paper electrophoresis, and spectrophotometry, washing experiments on hide powder which had been tanned with basic chromium chloride solutions having various acidities, and displacement of sulfato groups from chromium sulfate tanned hide powder with 2-4 molar sodium chloride solutions. I. D. C.

The Measurement of Leather. By A. Brill. *Das Leder* **3,** 299 (1952). Two types of machines, the pin wheel and the Sawyer, are used in Germany for measuring leather area. The pin wheel machine has measuring wheels one inch (English) apart from center to center and pins one inch apart in the wheel. It therefore measures accurately to one square inch. The Sawyer machine is less exact for three reasons. Thickness of the leather influences the measurements because a thick leather will raise the measuring wheel and start the measurement sooner than a thin leather. In all cases, the registering of area starts before the edge of the leather reaches the line connecting the axes of the measuring wheel and the driving wheel. There is also a wheel-breadth error because, on a slanting edge, the wheel is raised by the first part of the leather to touch the wheel. The recorded area is therefore high. The measuring wheel drives a ratchet wheel with pawl. On the average, at the end of the measurement, the ratchet wheel drops back one-half the distance between the teeth on the ratchet, thus giving low readings. I. D. C.

PATENTS

Modification of Keratin. U. S. Pat. 2,615,783. J. W. Haefele, Cincinnati, and N. B. Tucker, Glendale, Ohio, assignors to The Procter & Gamble Co., Ivorydale, Ohio. Appl. Aug. 11, 1950. In the process of treating keratin-containing substance to modify chemical and physical characteristics thereof wherein the said substance is subjected to the action of a sulfur-containing reducing agent and the disulfide linkages of keratin are converted to sulfhydryl groups, the step of reacting reduced keratin at a pH of about 4 to about 10.5 with an aqueous solution of at least one organic polythiosulfate of the group consisting of water-soluble polythiosulfates of alkanes and alkenes and substituted alkanes and alkenes

to convert reduced keratin to polydisulfide containing the organic group of said organic polythiosulfate.

Compositions for Treating Keratin-Containing Materials. U. S. Pat. 2,615,828. J. W. Haefele, Cincinnati, Ohio, assignor to The Procter and Gamble Co., Ivorydale, Ohio. Appl. Feb. 15, 1951. A new composition comprising an aqueous solution at pH 7 to 10.5 of the equilibrium reaction mixture of *(a)* water-soluble mercaptan of the general formula RSH and *(b)* water-soluble salt of thiosulfuric acid of the general formula $R'SSO_3H$ wherein each of the symbols, R and R′, represents a monovalent organic radical which is free of the concomitant presence of a carboxyl group and an amino group, and in which the free valence is on an aliphatic carbon atom, the total potential concentration of mercaptan in said solution being between 0.05 and 2.0 molal.

Tanning with Sulfonate Tannig Agents and a Formaldehyde-Arylamine-Cyanamide Type Compound. U. S. Pat. 2,621,102. D. E. Nagy, Stamford, Conn., assignor to American Cyanamid Co., New York N. Y. Appl. Dec. 6, 1947. A tanning process which comprises impregnating hides and skins capable of tannage with a solution of a sulfonate tanning agent and with a different solution of a water-soluble acid salt of a material obtained by condensing an aryl hydrocarbon monoamine selected from the group consisting of amines of the benzene and naphthalene series with formaldehyde and then reacting the condensation product so obtained with a member of the group consisting of cyanamide and dicyandiamide, said material containing from 2-11 guanyl substituted arylene radicals.

Process for the Treatment of Keratin and Related Materials. U. S. Pat. 2,622,036. P. Alexander and C. Earland, Leicester, England, assignors to Wolsey Limited, Leicester, England. Appl. Aug. 23, 1950. A process for the treatment of protein materials of high cystine content which comprises oxidizing a protein material of high cystine content with a solution of a saturated per-aliphatic acid having up to four carbon atoms in the molecule to disrupt the disulfide bonds of said protein material without substantially attacking the remainder of the protein molecule and dissolving at least a part of the oxidized product thus obtained in a solvent.

Thermosetting Keratinoid Compositions and Method of Making the Same. U. S. Pat. 2,623,039. E. Frankl, Rochester, N. Y., assignor to Rochester Button Company, Rochester, N. Y. Appl. May 2, 1949. Method of making a heat-hardenable molding composition which comprises subjecting a hoof meal to the action of an aqueous solution of a substance capable of splitting the disulfide linkage of the keratin of the group consisting of alkali metal bisulfites and organic mercapto compounds in the presence of a dispersing agent, and treating the resulting material in aqueous dispersion successively with phosphoric acid and a water soluble calcium salt.

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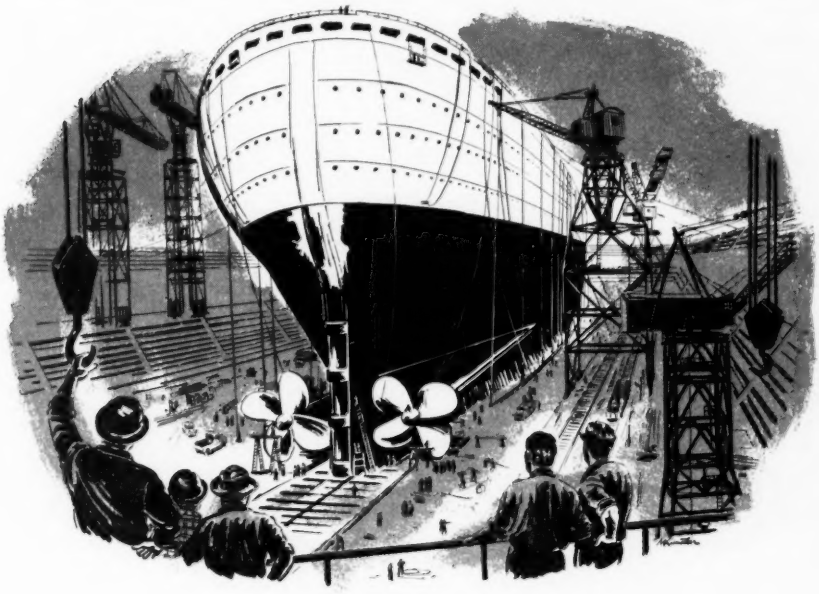

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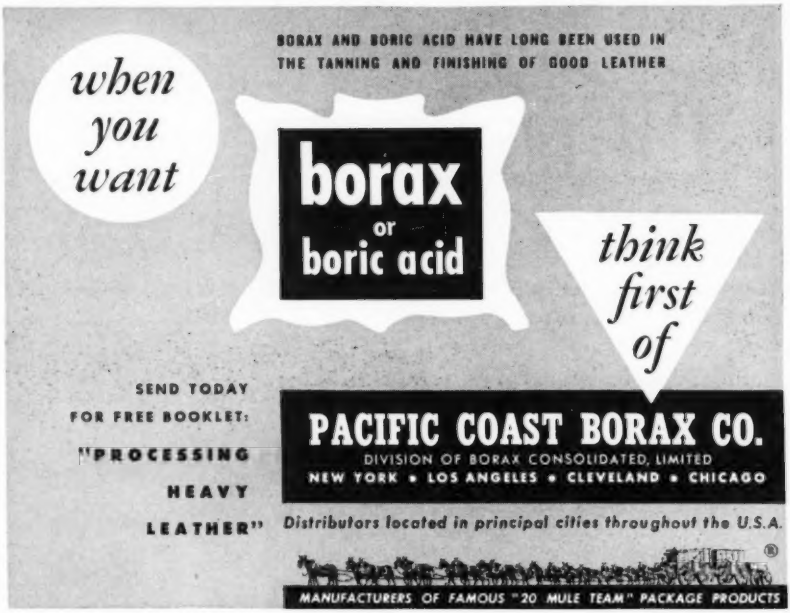
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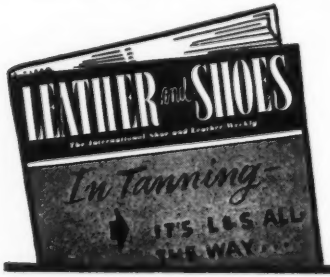

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WATER	15.98	11.5	6.7
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